Chemical Age

DU PONT POLICY IN WEST EUROPE
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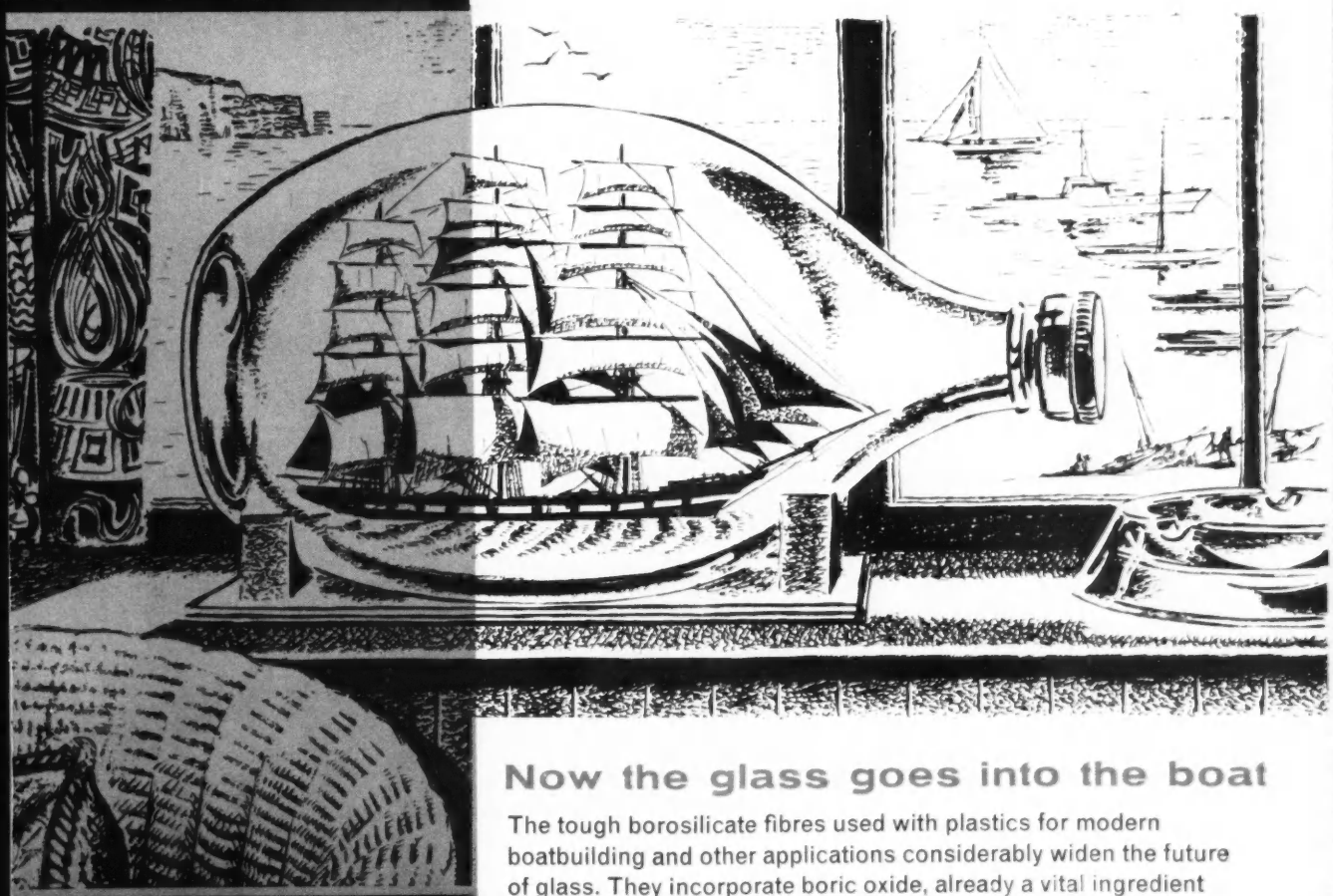
VOL. 81 No. 2079

16 May 1959

MORE IDEAS BEGIN WITH BORON



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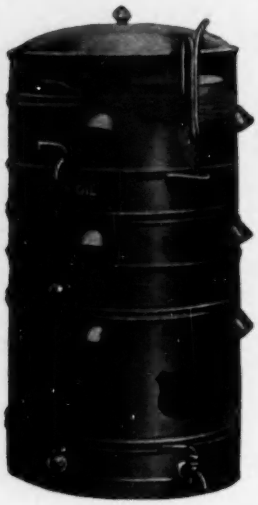

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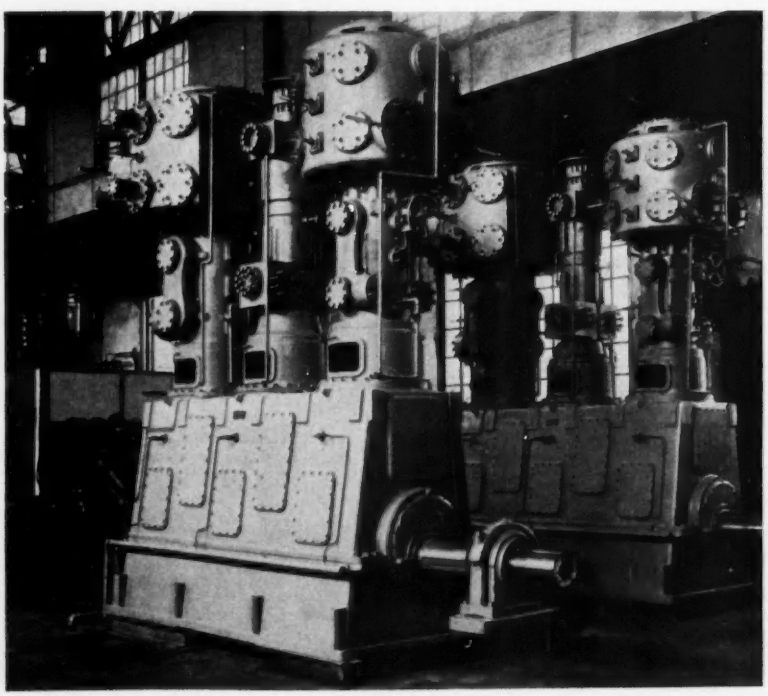

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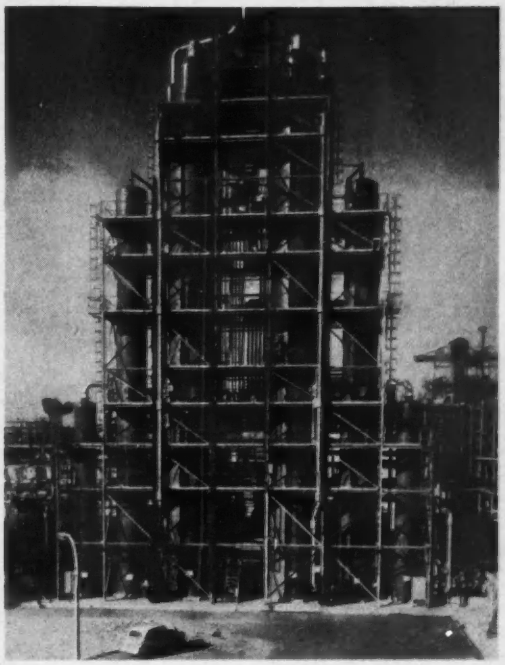
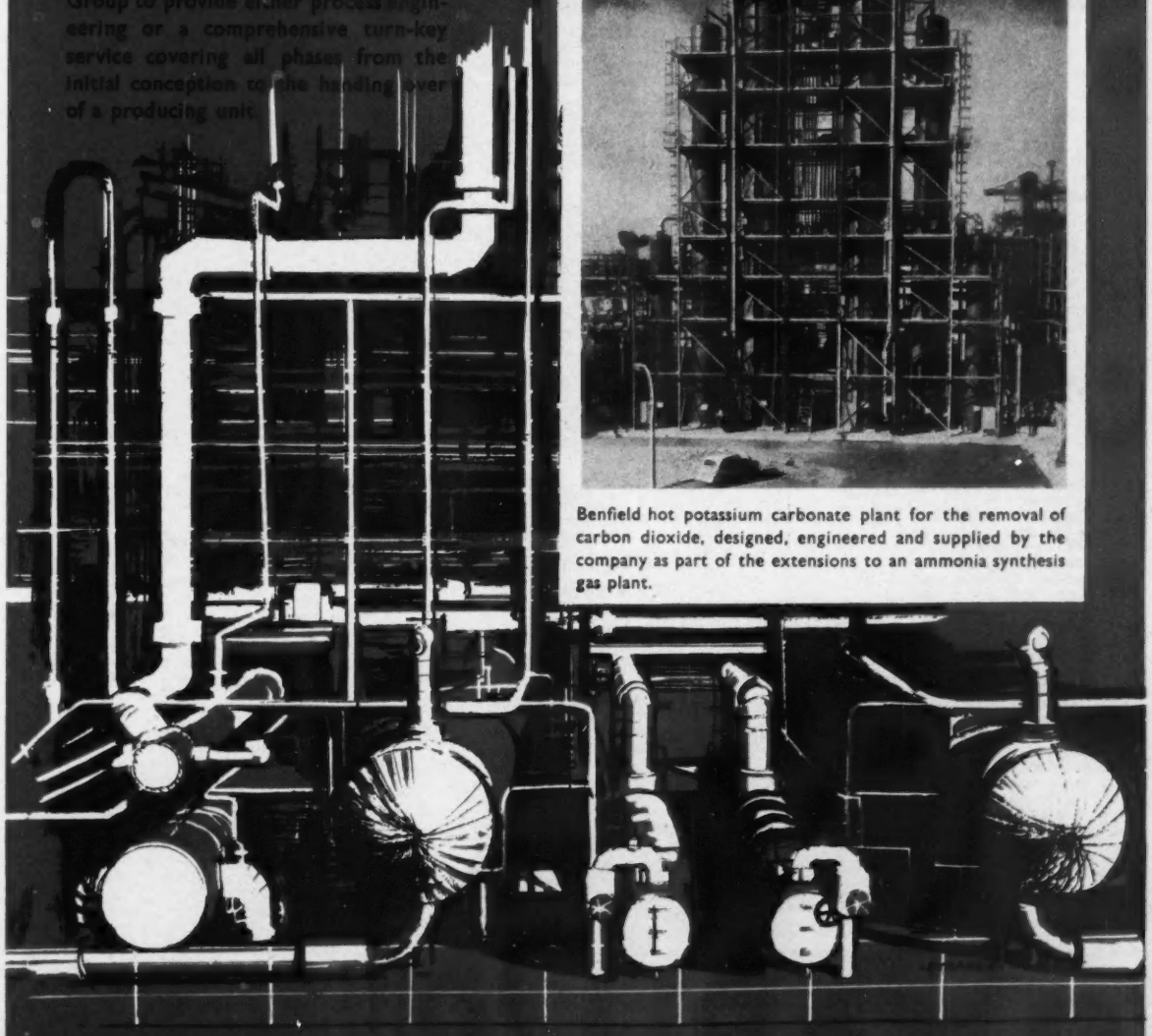


Benfield hot potassium carbonate plant for the removal of carbon dioxide, designed, engineered and supplied by the company as part of the extensions to an ammonia synthesis gas plant.



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Towers of Chemical Stoneware

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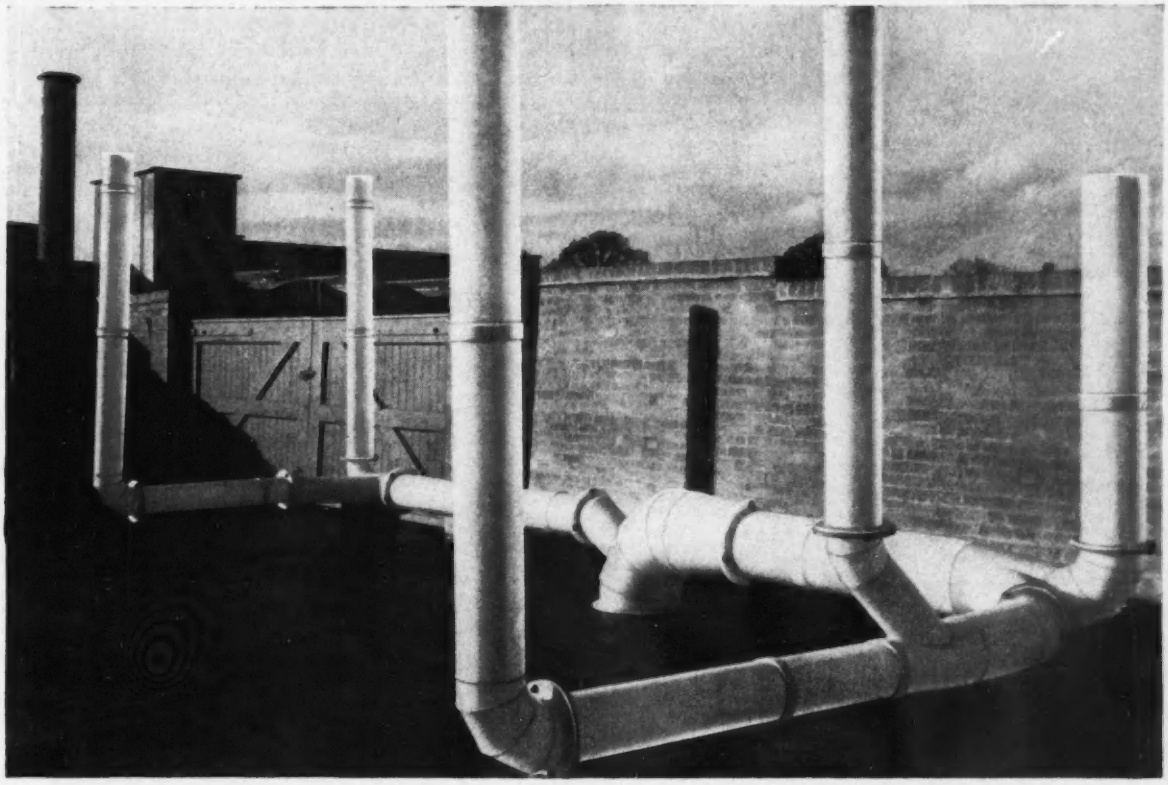
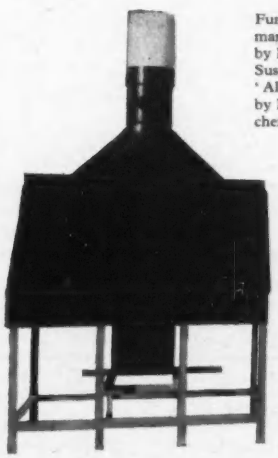


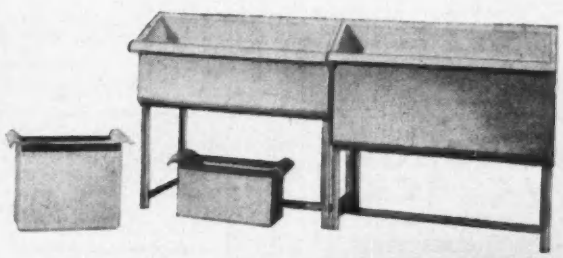
Chemicals will not corrode this fume exhaust plant —it is made from 'Alkathene'

REDIWELD LIMITED made this fume exhaust plant and manifold from ' Alkathene ' I.C.I. polythene, because ' Alkathene ' withstands chemical attack. It is strong and it will not crack. Chemical plant fume extraction systems made from ' Alkathene ' will last indefinitely. The sinks in the smaller illustration are lined with ' Alkathene ' as a protection against corrosive fluids.

' Alkathene ' is a light, easily fabricated material and is available in a wide range of colours.



Fume exhaust system and manifold made from 'Alkathene ' by Rediweld Limited, Crawley, Sussex.
' Alkathene ' lined sinks also made by Rediweld Limited for a chemical laboratory.



'Alkathene' is the registered trade mark for the polythene made by I.C.I.

'ALKATHENE'

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IMPERIAL CHEMICAL INDUSTRIES LIMITED · LONDON · S.W.1

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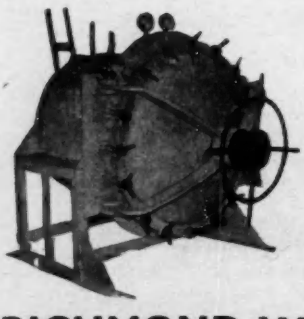

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IN STEEL
STAINLESS STEEL
ALUMINIUM
COPPER . ETC.

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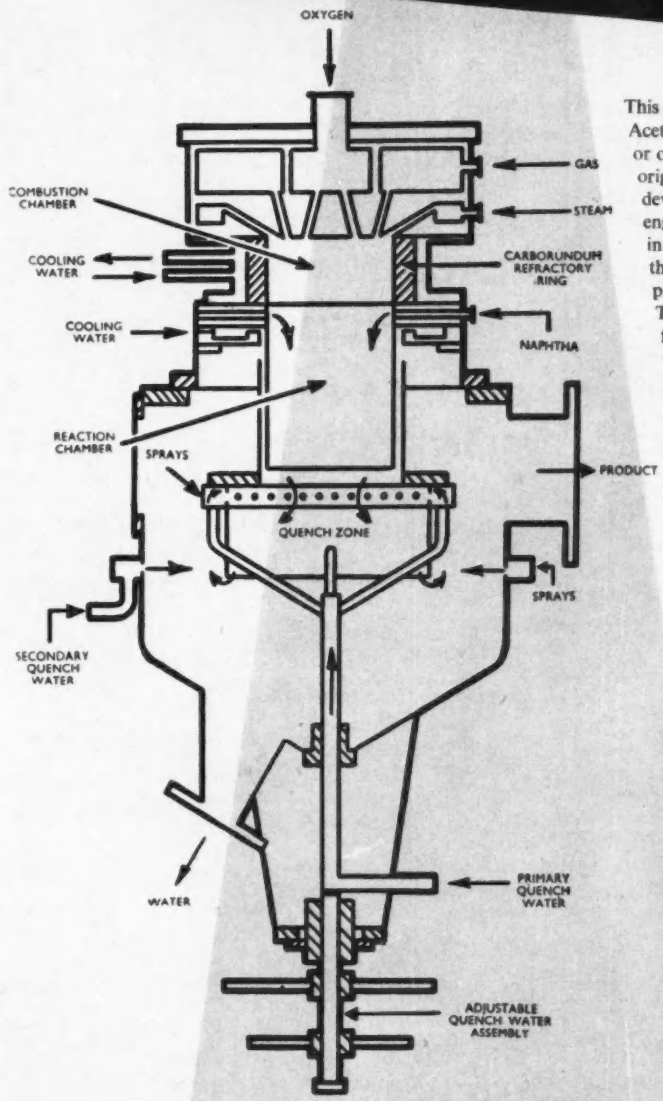

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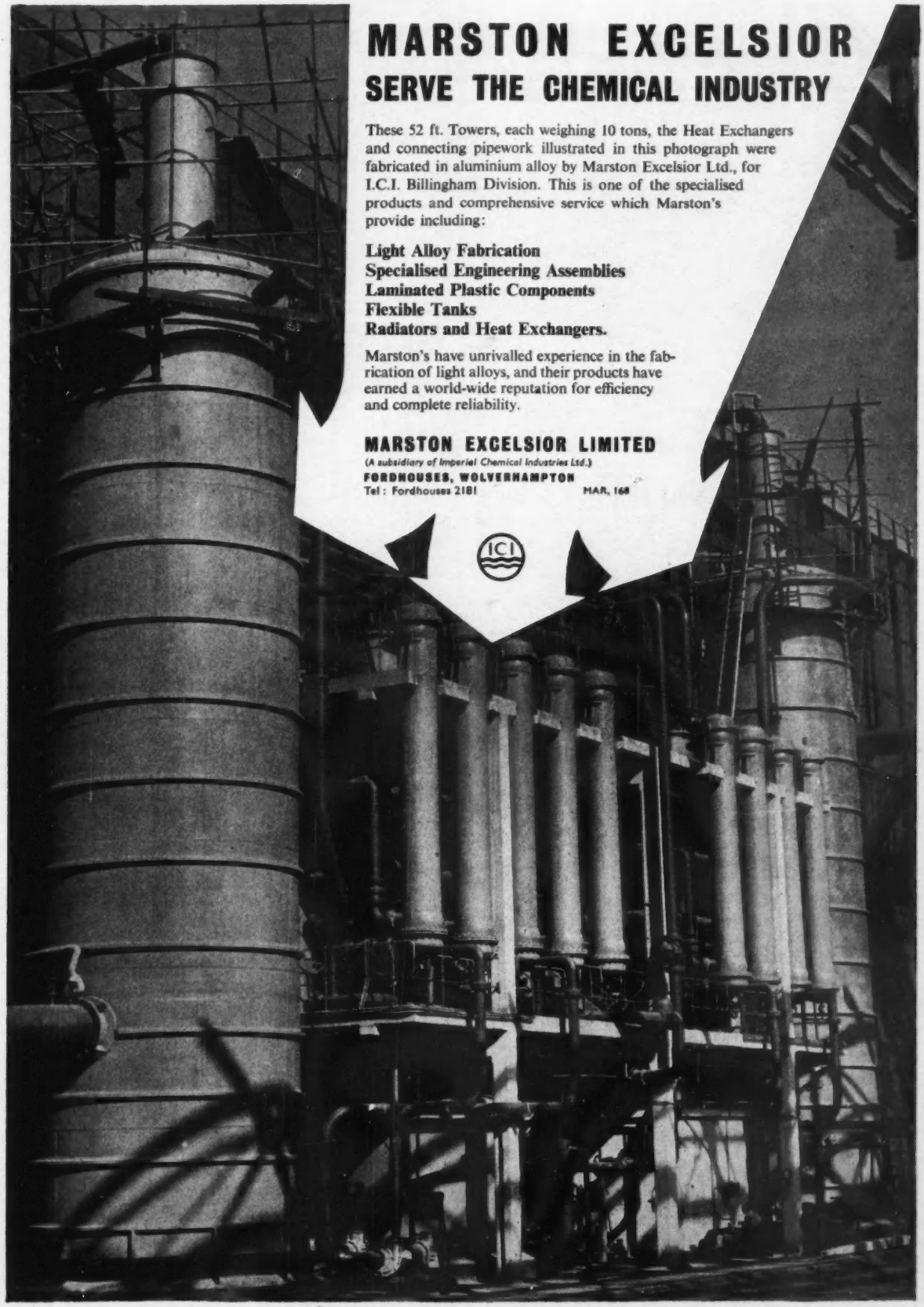

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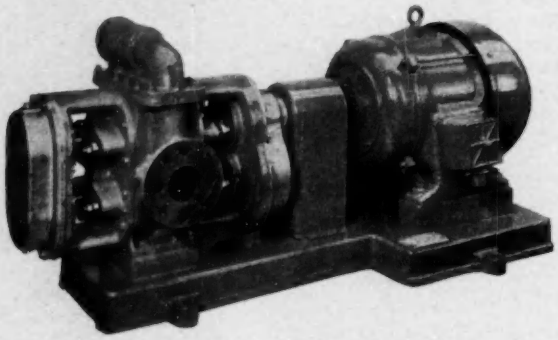

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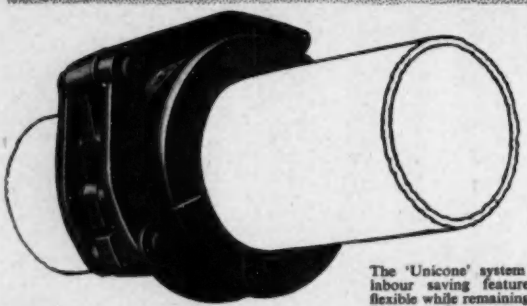

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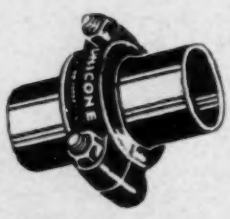

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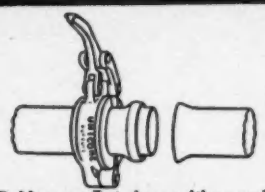
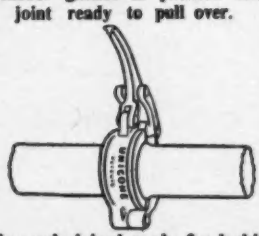


Rubber gasket in position and joint ready to pull over.



Pipe ends joined ready for locking.

The completed joint.

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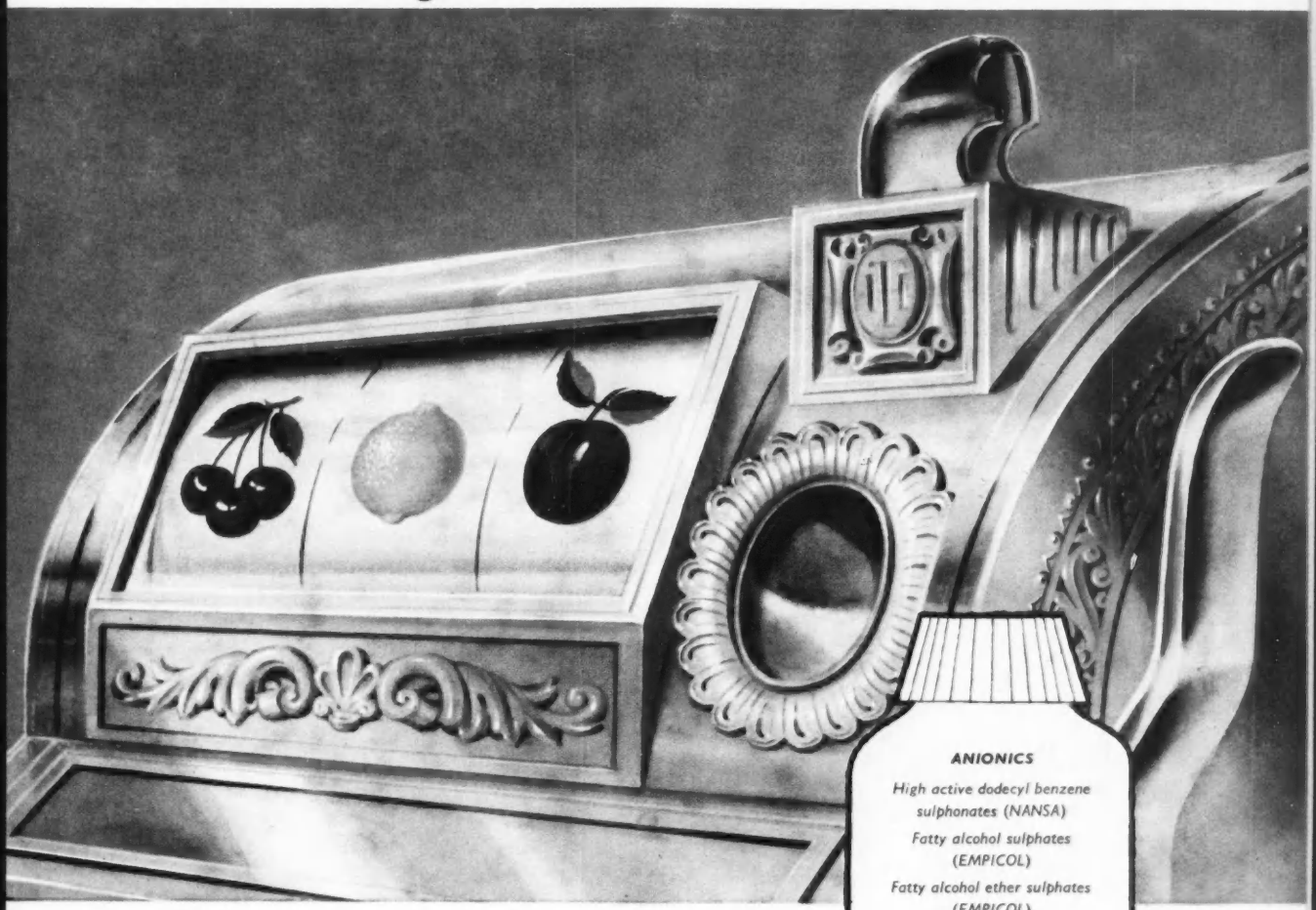

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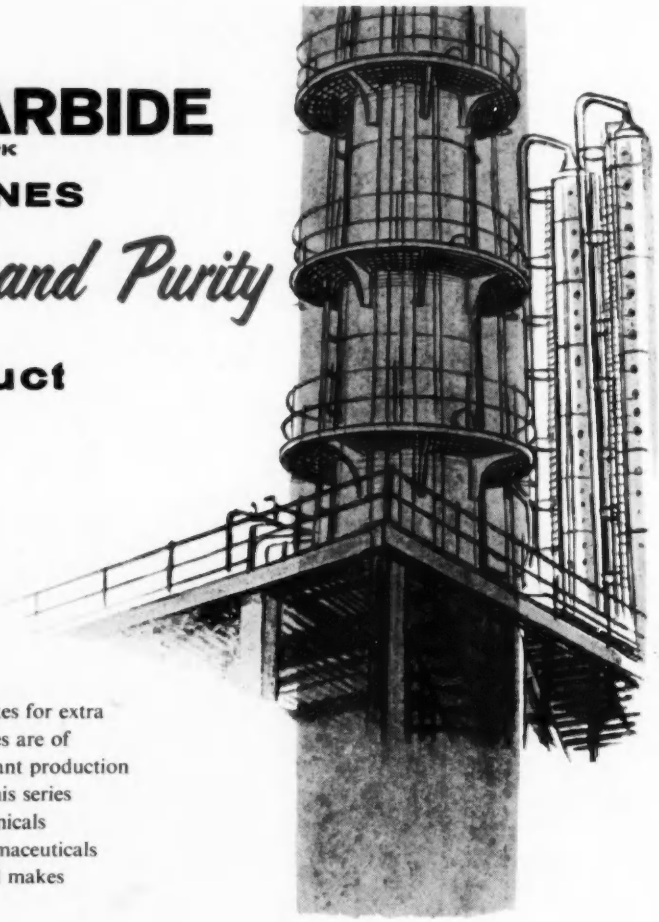

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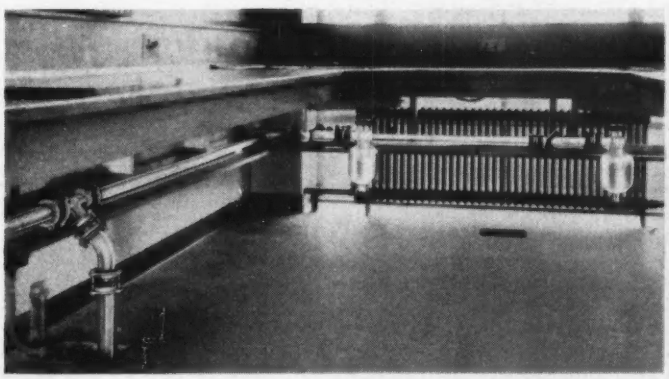

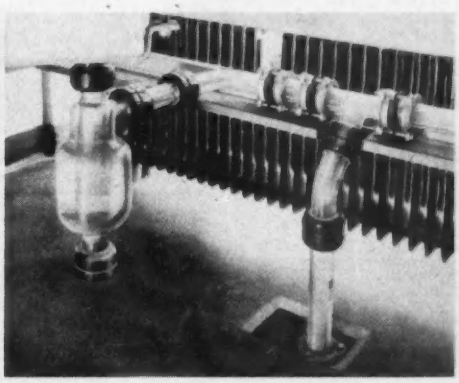

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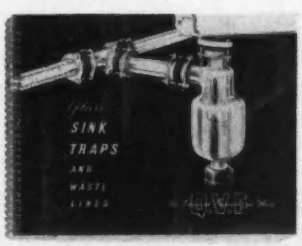

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CHEMICAL AGE

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RESEARCH AND D.S.I.R.

SINCE responsibility for the scientific direction of the Department of Scientific and Industrial Research was transferred to a research council drawn from outside the Council some two years ago, a number of decisions have been taken. In the main these have been concerned with the transfer or abandonment of work undertaken formerly by D.S.I.R. with the aim of making possible greater effort in directions considered to be of most value, or in utilising limited facilities to the best advantages.

In its second annual report (see also page 821) the Research Council, having had time to make a thorough survey of many aspects of D.S.I.R.'s work, now has a much clearer picture of the way in which the department should go. One of the main points made is that the Council has found that the amount of money spent on research at universities has not been adequate. The Council has therefore decided to increase its grants for special researches. The amount has already been increased and by the end of the second five-year plan, there should be a really worthwhile effort. This increase in grants to universities etc., is most satisfactory as is the Council's recognition that there should be financial support for longer terms than has been customary in the past to outstanding university investigators carrying out promising lines of research.

A new idea which it is believed will be of value is that D.S.I.R. is ready to consider proposals for the support of industrial research and development, whatever the scale, which is likely to lead to results of value to the nation. The Council feels that valuable ideas exist in some smaller industrial concerns in this country which they cannot afford to investigate and at the other end of the industrial scale there may be research and development projects that even large organisations cannot adequately finance.

Expenditure on revenue and capital grants to the research associations are expected to rise by about a third, because, as the last few years have shown, their scope and activities are growing steadily in response to the expressed needs of industry. The Council, however, points out that it is the policy of D.S.I.R. that industry should bear an increasing share of the total operating costs of these associations and it envisages that over the next five years grant-earning income from industry will be 46% greater than during the previous five-year period, the grants themselves only being 34% greater.

The grant-aided organisations in the Government scheme serve about 55% of British manufacturing industry. A wide variety of functions are performed, but the largest single item is undoubtedly applied research on problems common to each industry served. It seems that it is the Council's view that the various research associations have established themselves and are rapidly gaining that state whereby the increases both in scale of their work and the contribution science can make to the productivity of their member firms could lead to incentive gradually being reduced. Therefore the Council considers that any further expansion must be financed by industry (see CHEMICAL AGE last week, this page). Nevertheless, D.S.I.R. will continue to pay a block grant after this stage is reached, for by so doing it can exercise an important and beneficial influence on industrial

research, and can also ensure that each association has the necessary proportion of basic research in its programme.

Since the policy of reducing incentive grants was adopted in 1951 the overall ratio of grant to industrial income has fallen from 1:1.65 to 1:2.5 and the lowest individual ratio is now 1:4.6. But there is fortunately nothing mechanical about the trend. Each grant is fixed at each five-yearly review according to circumstances. Thus while terms in general are being stiffened, the total of all grants should increase to accommodate the normal growth of existing research associations, and the formation of new ones, over the next five years. A need for flexibility in reducing incentive grants is also being borne in mind. Indeed the Council emphasises generally in its report that it is keeping the budget for the five-year plan flexible because of the rapidly changing world conditions.

Another view expressed by the Council is that research associations should be encouraged to do a reasonable amount of sponsored research so long as co-operative research programmes do not suffer. This report's terms, general though they may seem, are along very much the right lines. Most actions taken to date have received favourable comment; the one action which we consider merits criticism is the disbanding of D.S.I.R.'s microbiological unit at Teddington. It will be of interest to see whether the universities will, in fact, make applications for grants with which to encourage new microbiological departments or strengthen existing ones.

CHEMICAL PUBLIC RELATIONS

ONE of the symposia at the American Chemical Society's 135th meeting was on chemical public relations and it is evident from the papers presented that although this country regards the U.S. as the leader in this field some chemical concerns there apparently do not have a grasp of the essentials of public relations. As the British chemical industry has, we believe, a genuine interest in setting up good public relations programmes we think that the comments and points made at the A.C.S. meeting will be of interest.

How to handle a disaster disastrously was dealt with by Henry H. Hunter, Olin Mathieson Chemical Corp. He said that the failure of company officials to establish a sound working relationship with the Press underlaid almost every instance of botching the public relations aspects of a plant accident.

Too frequently a disaster in a plant also becomes a disaster in public relations, and from a standpoint of the Press, disasters are mishandled. By far the biggest reason is the unwillingness of most managements to accept the fact that a disaster is anyone's business but its own. Hunter describes as tragic the first temptation of many executives to exclude the Press from the scene altogether (see CHEMICAL AGE, 7 March, page 395).

Principles offered as a guide in handling public relations in a disaster by Hunter are : Make one person responsible for handling the Press during an emergency; assign him this responsibility in advance. Anticipate questions the Press will ask; get answers promptly. Inform all news media as soon as possible; give them the name of a man to contact for information. Set aside a press room and provide telephone facilities. Arrange with plant guards and security people to get the Press into the plant.

What constitutes a good public relations programme in a disaster was also discussed by Arthur Northwood, Jr., Shell Chemical Corp., New York. This meant, Northwood said, permitting representatives of the Press, Radio and TV to find out as quickly as possible all the pertinent facts. These might include what happened, extent of damage, fatalities, cause, will the plant be shut down for long, etc?

The company that takes all reasonable steps to help the Press get the full story on the entire situation is much more likely to get sympathetic treatment than the company that does not. This also holds for off-site disasters, e.g., accidents occurring during consumer use of a company's products.

According to George M. Worden, Worden and Co., Washington 7, D.C., U.S. chemical industry deserves more credit than it is getting. Rockets and missiles, electrical and aircraft industries are getting the bulk of the glory in the U.S., and the same applies in the U.K. But it is the chemical industry which is providing the fuels, the man-made materials such as plastics and high-temperature lubricants, to make missiles and rockets possible.

Again, as in the U.K., there is a great deal of publicity about the high cost of health. Co-operative efforts between industry, trade associations and professional societies would be a big help here, considers Worden, for it would let the public know how vital the chemical industry is in peace and war.

If a company wants a good public relations programme, its executives must be thoroughly aware of their objectives, says W. Alec Jordan, of W. Alec Jordan Associates, 290 Park Avenue, New York. His criteria are: Ask key publications for their opinions and suggestions; re-analyse the news releases issued for the past few months; determine where public relations ranks in the organisation chart.

William Q. Hull, *Chemical and Engineering News*, states that every chemical company regardless of size needs a public relations programme. This it must devise to serve its specific needs. The results of a sound programme are felt when financing is desired, plant sites are needed, etc.

It is realised that the small company faces certain problems not felt by a large company. It has first to decide, for instance, whether to have its own public relations department or a public relations counselling company. It has to decide whether one person or department can handle advertising and public relations. A smaller budget also means a more difficult programme in deciding company activities, but even the less costly activities, says Hull, can result in satisfactorily establishing a small company in the eyes of the public.

Another interesting angle to public relations was suggested by Harry Levin, American Cyanamid Co., and Sydney Steele, Atlas Powder Co. Levin discussing 'Community relations in the chemical industry' reports that the benefits of effective community relations are threefold: attracting and keeping better employees and increased productivity, when the company is a desirable citizen; community understanding of the company's problems and needs; community relations help sales when neighbours are customers, and often shareholders.

Public relations plays an important part in personnel recruitment. Externally it can improve relations with the educational world by personal contact, distribution of published material, including annual reports, etc., and by publicising scholarship programmes and advertising employment opportunities. The Press is important because it is mainly through the printed word that a company becomes known externally; through co-operation with industrial relations, it can influence the internal climate so that present employees will think and speak well of the company.

Finally, how does a business publication evaluate a company's public relations department? William Olcott, of our U.S. contemporary, *Chemical Week*, deals with this question along these lines. One method of evaluating a company's department for an editor would be how fast they answer the queries of a publication. The public relations man should be easily accessible with the desired information on hand, states Olcott. If this is not possible the publication should be directed to the news source and given access to the executives involved in the story. Screening of calls by the public relations man's secretary and not answering for 2-3 days is a method to be avoided.

Du Pont's Policy of Expansion in Europe

Local Manufacture of Other Chemicals is Being Actively Studied, says U.K. Chairman

DU PONT policy envisages further chemical plants in the U.K. and West Europe and is aimed at making fullest possible use of local construction and operational labour, and local suppliers for plant, equipment and raw materials.

In the belief that there will be manufacturing opportunities in the U.K. and other parts of West Europe in addition to plants already in hand, the company is actively studying various possibilities for local manufacture of some of its other chemical products. Market studies are in hand on a number of products and each of the present sites, particularly that at Londonderry, where the neoprene plant is nearing completion on a 380-acre site, is large enough for sizeable expansion.

More than 87% of the equipment and supplies for the neoprene plant, valued at over £2.5 million, has been purchased in the U.K. The operational labour force of 400 will include only 18 Americans and this number will be reduced. In addition, bulk of the raw materials will be purchased in the U.K.

This important news of Du Pont policy so far as Europe is concerned was given at the new Elastomers Research Laboratory last week by Mr. David H. Conklin, chairman of Du Pont Co. (United Kingdom) Ltd. and international director in Europe of E. I. du Pont de Nemours, who spoke of the company's European plants in hand and of future plans, and Mr. William H. McCoy, managing director of the U.K. company, who gave the first progress report on construction of the neoprene plant.

Mr. Conklin said that the new laboratory, Du Pont's first completed unit in West Europe, was evidence of their plans to enter that market with manufacturing and research facilities and to "compete for the great business opportunities that exist here".

Other European Projects

In the three years since Du Pont set up the U.K. company, whose neoprene plant is due for completion early in 1960, three other West European subsidiaries have been announced. Du Pont de Nemours (Belgium) S.A. are building a new paint plant at Malines, which is expected to be completed by the end of this year. It will be the company's first manufacturing plant in the Common Market area.

Du Pont de Nemours (Nederlands) N.V. will start construction on a new plant for Orlon acrylic fibre at Dordrecht by the end of the year. A substantial part of the Orlon produced will go to the U.K., the biggest single export market for this fibre. The latest venture, Du Pont de Nemours (International) S.A., Geneva, is a full-fledged sales company to co-ordinate Du Pont's sales activities primarily in Europe for products made by the European subsidiaries and certain products from the U.S. parent company.

The company has well over 1,200 employees in West Europe, with the largest number in the U.K. Within a year, this number should be at least 1,500.

Mr. Conklin pointed out that the U.K. had for many years been one of Du Pont's largest single markets outside the U.S., just as the U.S. was a major export market for Britain. He hoped this relationship would be strengthened by the addition of local manufacture of the company's products which would mean jobs and opportunities for local people, products for U.K. export, better service to local customers and a stimulus for other businesses from which the Du Pont plant would make substantial purchases of raw materials and supplies.

D. H. Conklin

W. H McCoy

It was their firm policy to employ and train employees eventually to take over important management positions overseas. It was expected that the Londonderry plant would have about 18 American out of a total employment of some 400 and that those 18 would be reduced as rapidly as possible. There were great opportunities for local people to rise to top managerial posts in Du Pont's overseas companies. Mr. Conklin added "I know of no limitations on the advancement of any of our employees in the Du Pont Co. (United Kingdom) Ltd. who have the ability and aspirations to follow an industrial career."

Mr. McCoy declared that construction at Londonderry was about one-third complete. Barring unforeseen delays, production would start in the first half of 1960. The construction force totalled just over 1,000 and 98% of it had been employed locally. A great deal of the on-site engineering work was being handled by British experts. The monthly construction payroll was about £70,000 and more than 87% of equipment and supplies, valued at more than £2.5 million had been purchased in the U.K. Of the remaining 13%, 10% was purchased on the Continent and 3% came from the U.S.

He reported that the construction force had worked more than 1.5 million man hours without a serious disabling injury.

As the operating force took over in 1960 new jobs would be created for about 400 men and women, mostly from the U.K. About 20 men would by then have spent about a year each in supervisory training at the Montague, Michigan, works.

Despite the company's American origins, they felt that they had come a long way towards becoming a British company. Soon they would be buying their major raw materials for neoprene in the U.K., producing and selling in the U.K. with U.K. personnel.

He added that Du Pont were also in this country selling Orlon acrylic fibre, chemical weedkillers made by a British manufacturer, plus Butacite safety glass laminate.

New Elastomers Lab. is Du Pont's First Permanent Facility in Europe

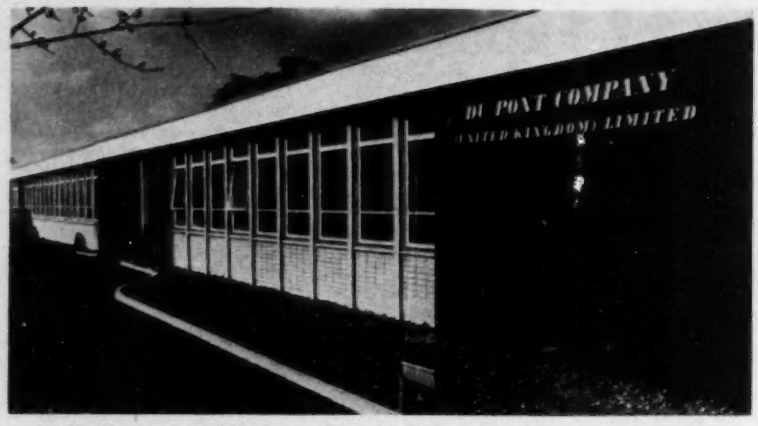
FIRST permanent Du Pont facility to be completed in Europe—the Elastomers Research Laboratory—was opened at Hemel Hempstead last week by the Du Pont Co. (United Kingdom) Ltd. With Dr. Wilfrid P. Fletcher, an authority on rubber, as manager, it will be staffed by British graduates and technicians.

This new modern laboratory, devoted to developmental work on neoprene, Hypalon and Viton synthetic rubbers and rubber chemicals for the European rubber industry, will be complementary to the Du Pont neoprene plant due on stream in Northern Ireland in 1960. It is expected that the service the laboratory will give will increase materially when the new plant is in full production.

Neoprene was first produced commercially by Du Pont in 1932 after nearly a century of research for a synthetic rubber which culminated in the discovery that monovinylacetate could be treated with hydrochloric acid to produce chloroprene. This material was soon converted to a rubber-like solid—neoprene.

Hypalon, the next Du Pont synthetic rubber, developed in the early 1940's, is made by reacting polythene with chlorine and sulphur, giving a vulcanisable rubber with many unusual properties. Latest addition is Viton, a fluoroelastomer, developed for service in oils, fuels, solvents and chemicals at temperatures over 400°F.

All three have found increasing application in industry, transport, engineering, clothing and in the home. Chemical



Du Pont's new Elastomers Research Laboratory at Hemel Hempstead

industry applications are many and include acid hose, tank linings, protective coatings, coated fabrics, sealants, etc. In addition Viton is used in fuel cells. All three synthetic rubbers offer good resistance to a wide range of chemicals, performance varying according to temperature, concentration of acid, etc.

For instance, little or no effect on neoprene is reported with 20% sulphuric acid at 200°F; the same is true of Hypalon with either 50% sulphuric acid at 200°F, 80% at 158°F or 95.5% at room temperature. Sulphuric acid, fuming, 75°F, when used with Viton results in 58% retention of tensile strength; 69% retention of elongation; a drop of 4 points in hardness; and a 4.8% increase in volume.

Two types of tests to evaluate the resistance of neoprene and the other synthetic rubbers to various chemicals will be undertaken at the laboratory. The first comprises immersion of elastomer samples in various chemical solutions and then measuring changes, if any, in the physical properties of the sample. The other method involves the application of coatings of various thicknesses to strips of metal that are then exposed to the chemical solution or vapour involved.

This electronic testing equipment can determine almost any of the physical properties of rubber samples from tensile strength to tear resistance and adhesive strength. It is highly instrumented and operates at temperatures ranging from −70 to 550° F.

The 10,000 sq. ft. laboratory is equipped with the most modern instruments and testing devices available from the U.K., Germany, France and the U.S.

There are three main parts: dry processing, fluid processing, and physical testing. In the dry processing area where solid rubber compounds are processed, one of the most unusual pieces of equipment is a high-pressure wire vulcanising unit. Simulating continuous vulcanisation conditions used in the wire and cable industry, it consists of a steam chamber for vulcanising at steam pressures of 100 to 275 p.s.i. and a water chamber for cooling at the same pressures. Before vulcanisation, an extruder covers the wire samples with compounds of neoprene or Hypalon synthetic rubber.

Paralleling the dry processing area of the laboratory is the section devoted to the processing of fluid elastomers such as those based on latex or solutions of rubber in solvents. This area is equipped with an ultrasonic homogeniser and a high-speed ball mill.

The physical testing area is equipped with an extremely accurate and complex electronic load-measuring tester as well as one of the latest types of weatherometers developed in Germany. The load-measuring equipment can be used to measure virtually any of the physical properties of rubber such as stress and strain characteristics, tear resistance, tensile strength, adhesion, and many others. It operates at temperatures ranging from −70 to 550°F. The weatherometer can duplicate almost any type of condition from intermittent rain and high humidity to dry or humid sunshine.

Other testing equipment in this section includes a salt-spray cabinet for problems relating to the use of maintenance coating of neoprene and Hypalon, a high voltage tester for determining the breakdown strength of wire and cable insulation, heat ageing ovens, flexometers, an R.A.E. freeze tester, a machine for testing the cracking resistance of neoprene soling compounds, abrasion and hardness testers, and an ozone cabinet.

Rumanian—I.C.I. Talks on Buying Terylene Know-how

TALKS are now taking place between Imperial Chemical Industries Ltd. and the Rumanian Government regarding the purchase of the 'know-how' of Terylene manufacture, Mr. G. Marcu, Commercial Counsellor at the Rumanian Embassy has announced. The supply of an ethylene plant is also being discussed with Humphreys and Glasgow.

Tenders have been asked for from British companies for a plant to extract oil from sunflower seeds.

U.K. Pharmaceutical Industry's Output Doubled Since 1948

SINCE 1948 the pharmaceutical industry's output and exports have more than doubled, with an increase in the labour force of under 10%.

In 1948 the industry, employing 48,600, had a turnover of £73 million, of which £15.8 million was exported; the provisional figures for 1957 show that the industry, employing 52,400, had a turnover of £150.4 million, of which £39.6 million was exported.

These figures are given in the Annual Report and Year Book 1958-59 of the Association of British Pharmaceutical Industry. The Association says that "there is a need to assemble facts about the provision and use of drugs, particularly the savings to the Health Service and the economy at large which the use of modern drugs produces". Today, 10 years after the inception of the National Health Service, manufacturers and wholesalers of pharmaceuticals receive less than 7% of total N.H.S. expenditure.

The report discloses that the chief overseas market was Australia (£3.3 million) followed by Nigeria (£2.1 million). Next came New Zealand (£1.9 million), the Union of South Africa (£1.8 million), India (£1.7 million), the Irish Republic (£1.6 million) and Pakistan (£1.5 million). In 1958 for the first time exports to the U.S. exceeded £1 million, and those to Canada were just under this figure.

The report reviews the work of the industry for the N.H.S., factors affecting pharmaceutical exports including the setting up of the European Common Market, and discloses that members of the association have informed the committee of the British Association for the Advancement of Science, under the chairmanship of Sir Hugh Beaver, that its members would welcome the adoption of the metric system in the U.K.

Shell Polypropylene for British Celanese Fibre

Polypropylene yarns are now being produced at the Little Heath, Coventry, factory of British Celanese Ltd. These yarns find their main use in industrial textiles.

Production is at the commercial development stage, but large-scale production is not being undertaken at the moment. It is understood that the polymer is being supplied by Shell Chemical.

SECOND D.S.I.R. 5-YEAR PLAN WILL EXPAND RESEARCH AND POSTGRADUATE PLANS

A MUCH clearer picture of the proper function of the Department of Scientific and Industrial Research has been reached, according to the annual report for 1958 of the Council for Scientific and Industrial Research (H.M.S.O., price 3s 6d net).

The Research Council, chaired by Sir Harry Jephcott, states that the second five-year plan has provided it with an opportunity of focusing its broad review of D.S.I.R. activities. It considers that the present expenditure is small in relation to the importance of its work, and that this expenditure should rise as more scientific and technological manpower becomes available and moreover, that expansion should not be tied too closely in advance to specific projects. The Council feels that while it is possible to estimate research needs fairly accurately two years ahead, it is only possible to give very provisional estimates for five years ahead. During the second five-year plan period, the Council has £61 million to spend compared with £36 million over the last five-year period.

As a result of these and other considerations, the department has reached a new decision about expenditure of research grants. The department, it is stated, is ready to consider support for industrial research which might lead to results of value to the nation. At the same time, the Council is embarking on a rapid expansion of grants for research and postgraduate training in the universities and other educational institutions. This means that the total number of studentships new and continuing should double—from about 1,900 in 1958-59 to 3,800 in 1963-64.

Three Kinds of Research

Even more rapid expansion is planned for special researches and longer financial support for longer terms is proposed for outstanding investigations of promising lines of research. Three kinds of research of importance to the nation are listed for D.S.I.R.'s research stations: research which helps central and local authorities to supply essential services and protect the welfare of the people; research which improves the efficiency of the economy as a whole; and research which provides industry with basic information for carrying out applied research.

Survey of present and projected research at D.S.I.R.'s research stations has continued, and confirmed the Council's view that careful selection of the most important projects is demanded.

Resources for the National Chemical Laboratory are to be concentrated fairly vigorously on a limited number of objectives. Three main categories of problem will be investigated. In the first are problems requiring researches of a fundamental nature which are of importance to the advancement of scientific knowledge and the development of industrial processes, and which are inappropriate to universities and other research organisations because they need special facilities, experienced staff and continuity of effort. In the second are problems the solutions of which are of considerable importance to many industries and to the national economy (e.g. control of corrosion of metals). The third category of problem is concerned with chemical researches for other organisations on repayment.

Because it is not felt that the N.C.L. has the manpower and facilities to make an important impact in the high polymer field, the Council has decided that in future the work on high polymers will be confined to research on polymer membranes, as used in electrodialysis, and to research on synthesis of resins for special purposes such as separation of metals.

Much of the work at Warren Spring Laboratory, Stevenage, which is being provided with facilities for pilot-scale investigations, will be essentially of a chemical engineering character. Mineral processing and synthesis of oil from carbon monoxide and hydrogen will receive special attention.

Changes at the Forest Products Research Laboratory are intended to give the laboratory more time for research that could lay the foundation for new developments. Hence certain responsibilities have been transferred to the Timber Development Association. The laboratory will begin research on pulping and other properties of home-grown timbers.

The transfer of the Ditton Laboratory and the Low Temperature Research Station to the Agricultural Research Council has been agreed together with that of the Pest Infestation Laboratory: it will take effect from 1 July. The only part of the Food Investigation Organisation to remain with D.S.I.R. will be the Torry Research Station for which a special steering committee has been set up.

Market for U.K. Creosote in British Columbia

A good market is offered to U.K. manufacturers of creosote in British Columbia, a U.K. Trade Commissioner Service market digest states. The Eastern Canada coal tar industry can supply almost 80% of Canadian requirements, but British Columbia is too distant for economic supply by rail from Eastern Canada.

Creosote is the most extensively used wood preservative in Canada, but proprietary formulas sold in concentrated form are also supplied by domestic production and imports from the U.K., U.S. and Sweden.

Sulphuric Acid Consumption Remains Level

CONSUMPTION of sulphuric acid in the U.K. at 615,480 tons for the first quarter of the year was little changed from the 615,324 in the corresponding quarter of 1958.

Production and consumption figures are:

SULPHURIC ACID AND OLEUM
(*Tons of 100% H_2SO_4*)
1 January to 31 March

	Chamber and tower only	Contact only	Chamber, tower and contact
Stock, 1 January 1959	22,209	76,263	98,472
Production	112,426	499,230	611,656
Receipts	22,863	12,242	35,105
Oleum feed	—	330	330
Adjustments	—	+213	+213
Use	80,832	306,135	386,967
Despatches	53,693	203,279	256,972
Stock, 31 March 1959	22,973	78,864	101,837
Total capacity represented	148,310	573,990	722,300
Percentage production	75.8%	87.0%	84.7%

Note.—These summaries exclude all Government plants.

CONSUMPTION
1 January to 31 March

Trade Uses	Tons 100% H_2SO_4 1958	1959
Accumulators	2,943	3,103
Agricultural purposes	273	298
Bichromate and chromic acid	5,163	4,496
Bromine	3,247	3,882
Clays (Fuller's earth, etc.)	2,430	2,901
Copper pickling	547	614
Dealers	2,774	3,549
Drugs and fine chemicals	5,352	4,616
Dyestuffs and intermediates	20,383	21,203
Explosives	3,455	2,478
Export	1,262	768
Glue, gelatine and size	157	112
Hydrochloric acid	14,393	12,514
Hydrofluoric acid	3,026	3,182
Iron pickling (incl. tin plate)	30,801	28,189
Leather	1,123	1,130
Lithopone	2,641	3,026
Metal extraction	745	692
Oil refining and petroleum products	14,251	15,214
Oils (vegetable)	2,812	1,938
Paper, etc.	1,394	1,776
Phosphates (industrial)	174	538
Plastics, not otherwise classified	9,775	10,355
Rayon and transparent paper	65,208	66,091
Sewage	2,518	2,879
Soap, glycerine and detergents	28,931	32,774
Sugar refining	138	126
Sulphate of ammonia	81,941	71,895
Sulphates of copper, nickel, etc.	4,649	7,952
Sulphate of magnesium	44	35
Superphosphates	155,114	163,600
Tar and benzole	8,534	7,324
Textile uses	2,796	2,658
Titanium dioxide	87,482	85,999
Unclassified	48,848	47,573
Total	615,324	615,480

RAW MATERIALS
Tons

	Pyrites	Spent oxide	Sulphur: Imported	Sulphur: Recovered, H_2S and filter cake	Zinc concentrates	Anhydrite
Stock, 1 January 1959	186,241	97,515	75,710	6,337	92,229	11,160
Receipts	48,953	53,872	71,635	13,926	32,647	189,647
Adjustments	+2,606	+3,994	+232	+856	+295	—585
Use	83,853	56,827	81,117	13,725	45,778	189,338
Despatches*	1,735	905	930	8	—	—
Stock, 31 March 1959	152,212	97,649	65,530	7,386	79,393	10,884

* Including uses for purposes other than sulphuric acid manufacture.

★ OUTCOME of the visit of Sir David Eccles and his trade mission to Russia will be awaited with interest by the British chemical industry. The Russian market is, on paper, a potentially valuable export outlet for our chemical producers, most of whom have exportable surpluses of one kind or another.

But it has been made abundantly clear that the Soviet Union is more interested in selling chemicals here than in buying British products. U.K. manufacturers of chemicals and chemical plant have long since learned to discount as political hooha Soviet talk of trade with the U.K. in terms of £multi-millions. Experience with visiting missions from Russia as well as regular contacts with the Soviet Trade Delegation at Highgate has proved that the Russians are more interested in finding out process details than in buying either chemicals or plant.

The only sales to the Soviet Union are likely to be in process know-how—where they cannot find this out for themselves—and contracts for the construction of complete plants. Trade in both these highly valuable commodities is not likely to be large numerically although a few spectacular contracts will be signed in the coming years. Unfortunately there is no repeat business in either.

At present the U.K. chemical industry has no collective policy on trade with Russia. As trade can be undertaken in practically all chemicals, the U.S.S.R. is looked on as a normal export market, with trade policy the concern of individual companies. At any rate, I do not expect to see any large increase in shipments of British chemicals and plant items to Russia.

★ THE days of the soap war are numbered. Nowadays it is the syndet clash with no holds barred. Tide's new magic ingredient gets clothes cleanest; New Tide with Bluinite, a remarkable new discovery exclusive to Tide; only Daz has the blue whitener—an exclusive formula; new Surf washes shining white—it has double whitening power; Omo adds brightness to whiteness; Fab's fabulous new ingredient for whiter, brighter washes; Blue Spel—washday's magic wash.

But which really washes whitest? The B.S.I. Consumer Advisory Council in 'Shopper's Guide' concludes that all syndets are "much of a muchness, and the best value is whatever brand may be offering a reduced price at the moment". Such cynicism in the face of all those claims!

My readers will have little difficulty in identifying the common denomination for all those 'exclusive' ingredients that give whiter whiteness: they are the fluorescers, or optical bleach, which are colourless dyes that adsorb u.v. light from daylight and emit a bluish-white light. They neutralise the yellowish tint of a white fabric, making it appear even whiter.

The chemical industry is also responsible for most of the other ingredients that go into the syndet packet according to the council's analysts. The basic material, of course, is alkyl aryl sulphonate, but I was surprised at some of the other ingredients added in varying degrees by the manufacturers. These have been separated by the council's analysts.

Sodium polyphosphate aids active ingredient to remove dirt; *sodium sulphate*—a convenient filler; *sodium silicate* helps remove and emulsify fatty acids and *sodium carbonate* with a similar washing effect; *sodium chloride*, which "so far as is known has no purpose"; *carboxymethylcellulose*, soil-suspending agent; *EDTA* protects some ingredients from impurities in the water; *perborate*, oxidising bleach and stain remover.

★ I AM glad to welcome Mr. H. M. Arnold, acting managing director of Bowmans Chemicals Ltd., as a supporter of CHEMICAL AGE's campaign for more information from the British chemical industry.

At a luncheon on his company's recent open day—when visitors saw the plant for lactic acid purification by continuous solvent extraction—he drew attention to CHEMICAL AGE'S comments on secrecy in the chemical industry and assured the journalists present that it was Bowmans' policy to give them full information about their processes.

Of course they feel that as the world's leading producers of lactic acid and lactates they have nothing to fear. But it is often leaders in a particular field who are anxious not to run the slightest risk of giving the least little thing away.

★ SHADES of Hercule Poirot! After three years of scientific detection work, starting with no clue whatever, one of Teesside's most notorious offenders has been tracked down. In 1955 when an extremely objectionable 'tom-cat' smell was first reported, accusing fingers were pointed at I.C.I. Billingham. Nothing made at Billingham had the smell, but it was not long before the investigators were 'led' to the acetone plant where it was found that the water in the hydrogen gasholder had an unpleasant smell.

Confusion arose when the smell was reported during a period of shut down of the acetone plant. Then came the second clue. A laboratory sample from one of the plant's drains developed a distinct cat smell after it had been standing in the absence of air. It was found to contain bacteria which in the absence of air fed on something in the water producing hydrogen sulphide. After much research it was discovered that one of several chemicals present in a sample of effluent was the source of the trouble. Ironically this chemical has a sweet peppermint smell.

It was also found that in warm weather a section of the Tees is subject to gross pollution; water temperature is high enough and oxygen content low enough to support the bacteria identified in the laboratory. That explained why the offending smell hardly ever appeared in the wetter and colder months.

Final link in the enquiry was to destroy the chemical which gave rise to the smell. It was discovered that chlorination of the effluent would do the trick and so a plant is now in hand at a cost of about £40,000.

Clearly, Teessiders whose nasal organs have been assailed by industrial odours for many a year had something to complain about if hydrogen sulphide was in the air. But at least Billingham was only partly to blame and even Poirot would have said that honour has been satisfied.

★ CHEMICAL industry executives are among the most travelled in British industry. They have the example of Sir Alexander Fleck who probably covers as much 'ground' in the course of a year as the former American Secretary of State. On his recent tour of India and Pakistan, Sir Alexander never got to Kalimpong, that little hill town described by Chinese Communists as the "Trigger spot of the Tibetan revolt".

Neither was Kalimpong a scheduled stop for two Albright and Wilson directors on their recent extensive tour of India. But Mr. John Christopherson, director of Albright and Wilson Ltd., and Mr. L. W. Stubbs, sales director of Albright and Wilson (Mfg.), could not resist an invitation to escape the heat of Calcutta to visit a house on the outskirts of the town.

Although Kalimpong will not feature in their report on the sales potential of the Indian sub-continent, it was a visit that neither will forget. The town at the foothills of the Himalayas was full of Tibetans for it is the Indian terminus of the ancient Lhasa trade route. While visiting a Tibetan monastry in the district, they were told that a few days before some 2,000 mules had camped nearby. They were part of a large Tibetan mule train that had brought "some million pounds worth of gold into India under bundles of yaks wool". The monasteries were taking good care to see that some of their treasures did not find their way to Peking.

Alembic

New Route to Associateship Of Plastics Institute

THE council of the Plastics Institute has included in its revised regulations for the associateship (A.P.I.) a new route without examination open to candidates holding suitable qualifications in appropriate basic subjects, who have acquired knowledge of plastics technology and science as a result of satisfactory experience in the plastics industry.

There is a record number of 133 candidates for the 1959 examination to be held in a number of centres from 8-13 June.

Registration forms are now available from the Borough Polytechnic, Borough Road, London S.E.1, for the teachers' course on welding of plastics to be held at the Polytechnic from 29 June to 10 July.

Lord Mancroft will be the guest of honour at the Institute dinner to be held at Quaglino's on Friday, 26 June. Sir Miles Thomas and Mr. A. A. Part (Under Secretary, Ministry of Education) will also speak.

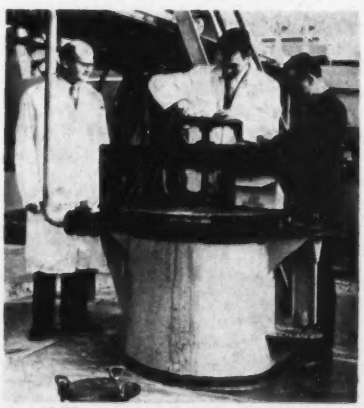
I.C.I. Supply Para-xylene in Bulk to Hoechst

I.C.I.'s Heavy Organic Chemicals Division's olefin works at Wilton, Yorks, has delivered a consignment of several hundred tons of para-xylene by water to Farbwerke Hoechst of Frankfurt on Main.

Hoechst are licensed to produce a fibre of the Terylene type from this material, but had previously obtained their para-xylene from the U.S.

This was the first bulk shipment by sea. The para-xylene was loaded into a ship at Teesport and taken to Dordrecht, Holland, where it was transferred to a Rhine barge for the rest of the journey to Frankfurt.

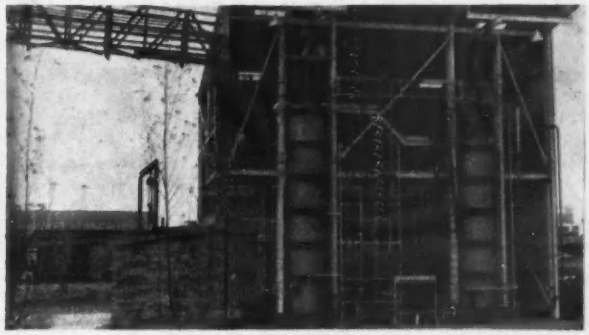
As para-xylene solidifies at temperatures below approximately 55°F, steam



An ICI representative supervises the transhipment of the para-xylene consignment at Dordrecht

was kept in continuous circulation in coils in the ship's tanks to keep it liquid throughout the voyage. Insulated tankers took the para-xylene by road from the works to the port.

BOWMANS PIONEER NEW PROCESS FOR LACTIC ACID



Extensive use of glassware is seen in this picture of the new lactic acid solvent extraction plant at Bowmans' Widnes works. Instruments are housed in the brick structure on the left

LACTIC acid is being produced by a new method based on solvent extraction at the Widnes works of Bowmans Chemicals Ltd.

At the same time the company has published results of research into the effects of calcium lactate on egg production. It is claimed that the addition of 1% calcium lactate to laying rations gives increased egg production, combined with reduced consumption of food and an increase in the weight of the hens.

It is also claimed that calcium sodium lactate is effective in preventing and curing cattle acetonaemia and twin-lamb disease.

Lactic acid is produced by the anaerobic thermophilic fermentation of sugars. The product is a dilute calcium lactate solution containing those complex impurities which invariably result from a fermentation process. The conventional purification process involves a sequence of coagulation, decolorisation, crystallisation, acidification, removal of heavy metals, and evaporation under vacuum, a number of the stages being repeated. The reason for this complicated sequence is that lactic acid is an involatile liquid with a great affinity for water, which forms no highly insoluble salts. Lactic acid is also extremely corrosive to nearly all metals and alloys. It has hitherto been a somewhat costly compound in the pure state.

The new process is based upon solvent extraction. When its development on the large scale came to be worked out it was realised that the change from batch to continuous working which it involved would enable other new purification techniques to be introduced. In the event, the solvent extraction plant, devised by Bowmans' technical manager, Mr. C. G. Childs, M.Sc., A.R.I.C., is merely part of a vastly improved purification procedure.

The dilute calcium lactate from fermentation, after removal of bacterial protein, is acidified and calcium is removed as the sulphate. The dilute lactic acid is then decolorised in activated carbon columns, which are regenerated in sequence. Regeneration of carbon of this type is thought to be entirely new, and, together with other features of the process, has been patented.

The dilute acid is then freed from residual calcium, amino-acids and traces of heavy metals by percolation through ion-exchange resin, which is regenerated in the usual way.

The purified acid is then evaporated under vacuum to high concentration and the product is extracted in counter-current with an organic solvent. The enriched solvent is then re-extracted in counter-current with distilled water and the stripped solvent is re-circulated to the extraction tower.

After a final decolorisation and ion-exchange treatment the pure aqueous extract is ready for sale or further evaporation to the strongest grade.

From the extraction tower onwards the acid is handled only in glass, stoneware, plastic or rubber-lined equipment.

This is believed to be the first purification process by continuous solvent extraction. It will work largely automatically, needing only a man to take periodic readings in the control room.

The new plant is designed to produce the equivalent of 2,000 tons a year pure lactic acid. The actual product goes out at 44 or 80%.

Aluminium for Sub-zero Methane

THE part played by aluminium in the bringing to the U.K. of the first shipment of liquid methane in the *Methane Pioneer* on 20 February, is shown in a film 'Aluminium for sub-zero methane,' made by the Aluminium Development Association.

The film refers briefly to low-temperature tests on aluminium alloys, as a result of which five tanks of 400-ton capacity were fitted into the *Methane Pioneer*. It then deals with the fabrication of two 1,000-ton aluminium alloy storage tanks, one by the A.P.V. Co. Ltd., and the other by Whessoe Ltd., and two 2,000-ft. aluminium alloy pipe lines running from tanks to jetty.

An important aspect was frequent testing and inspection to avoid risk of leaking liquid methane. Radiographic inspection of welded joints was carried out.

Acrylonitrile Copolymers Fibres May be Spun with N-nitroso Piperidine as Solvent

A NEW organic intermediate now available from Robinson Brothers Ltd., Ryders Green, West Bromwich, in evaluation quantities and suggested as a solvent from which fibres made of acrylonitrile copolymers may be spun is N-nitroso piperidine. Besides being used as an intermediate for the formation of N-piperidyl hydrazine, N-nitroso piperidine has also been suggested as a stabiliser in halogenated hydrocarbons against decomposition in electric capacitators.

This is one of a number of new products on which new literature has been issued by Robinson Brothers. The other products include two organic intermediates (N-methyl piperidine and methylene dipiperidine); carboxymethylmercapto-succinic acid, the first of a series of six thioether dicarboxylic acids available in trial quantities for use as metal deactivators; monoethanolamine thioglycollate, now in production and an alternative to ammonium thioglycollate; and N-ethyl piperidine, also in current production, and used in the purification of penicillin.

N-nitroso piperidine is miscible in all proportions with benzene, ether, ethyl acetate, acetone, carbon tetrachloride and ethanol. It is soluble in concentrated hydrochloric acid, but is reprecipitated on the addition of water.

The nitroso group can be reduced to an amino group if the reduction conditions are not too severe, with the formation of an unsymmetrical hydrazine-N-piperidyl hydrazine b.pt. 146°C/728 mm. This material is monobasic (hydrochloride m.pt. 162°C) and forms hydrazides when reacted with aldehydes and ketones. N-nitroso piperidine can be reduced to the hydrazine with zinc and 50% acetic acid, lithium aluminium hydride, sodium and ethanol, and hydrogenation under pressure with noble metal (Pd, Pt) catalysts.

Treating N-nitroso piperidine with zinc and hydrochloric acid or sodium amalgam leads to hydrolysis with the formation of piperidine. Piperidine is also formed on treating the product with excess concentrated hydrochloric acid for five hours, or on passing hydrogen chloride through a boiling solution in toluene for 10-20 minutes. It is comparatively stable to alkaline hydrolysis, 75% being recovered after refluxing with 20% excess caustic soda after 15 hours.

Electrolysis in sulphuric acid solution leads to the formation of what has since been proved to be isotripiperidine, m.pt. 97-98°C. At low temperatures, addition compounds are formed by ni-nitroso piperidine and dinitrogen tetraoxide in 1:1 and 1:3 molar ratios.

For methylene dipiperidine, with carbon disulphide-methylene piperidinium pentamethylene dithiocarbamate, m.pt. 58-59°C—has been used as a rubber accelerator. Methylene dipiperidine has also been suggested as an anti-rust additive in turbine oils (U.S.P. 2,388,058 and 2,388,059).

The other new organic intermediate, N-methyl piperidine, has as its chief uses the formation of quaternary salts by reaction with an alkyl halide, or with esters of iodo-or chloracetic acid; oxidation with hydrogen peroxide to give N-methyl piperidine oxide; reaction with sulphur dioxide to give an amine bisulphite complex; on electrolysis in an hydrofluoric acid solution all the hydrogen atoms are replaced by fluorine atoms with the formation N-trifluoromethyl decafluoropiperidine, said to be an inert liquid suitable for refrigerants or heat transfer media.

Other uses are as a basic catalyst, the separation of isomeric acids by fractional crystallation of their N-methyl piperidinium salts, e.g., isophthalic and terephthalic acids.

Carboxymethylmercapto-succinic acid and its esters have been suggested as metal deactivators to inhibit the metal catalysed deterioration of edible oils. The esters have been suggested as anti-corrosion inhibitors in lubricating oils, as synthetic lubricants and as textile auxiliaries.

Uses for N-ethyl piperidine are as an organic intermediate, the purification and separation of penicillin, particularly penicillin G; while the adduct with sulphur trioxide has been suggested as a reagent for sulphonating the leuco compounds of vat dyestuffs and other hydroquinones.

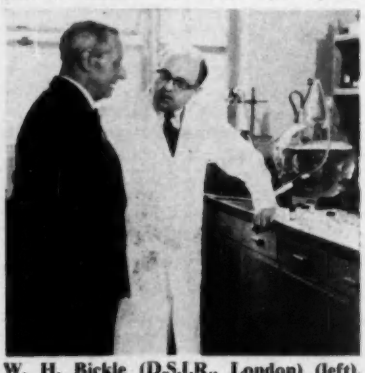
B.R.R.A. Hope to Modify Cellulose During Growth to Give Crease-resisting Cottons

AN investigation of the formation of cellulose is an intriguing section of biological chemistry now being undertaken at the British Rayon Research Association, and demonstrated at the Open Days on 6 to 8 May.

Work began first with the flax plant. In the laboratories, however, it has been found difficult to obtain a continuous supply of plants of any sort so that they have looked around for another series of cellulose which can be used chemically.

It is now possible for the laboratories, on the basis of the work done, to obtain an idea of how the cellulose is built up in nature. To determine what goes on it was decided to dissect a bacterial cell. Investigation is also taking place of the sugar phosphate metabolism. As a result of the work now being done in the biology section, it is hoped that the structure of the cellulose may be modified during growth, as the plant produces it.

"If we can feed the plant in a different way we may get a different cellulose," said an official. In other words, he added, they were hoping to grow plants which would produce crease-resist cotton!



W. H. Bickle (D.S.I.R., London) (left), talks to Dr. J. A. Gascoigne (British Rayon Research Association) beside a display demonstrating attacks of fungi on dyed viscose at the association's open day

Degradation of cellulose and cellulose cross-linking are examples of other work which is going on at the present time in this department.

The water sorption of textile materials is other work which is being done in another section.

Heavy water, deuterium oxide D_2O, is used in the water sorption experiments in place of H_2O in the region accessible to water (and thus to heavy water). All the hydroxyl groups are deuterated (i.e. become OD groups) and the surplus D_2O is now dried out of the sample leaving the accessible regions 'labelled' with OD groups. The number of these OD groups in the sample (and hence the fraction of the OH groups in the original material which are accessible to water) is determined by 'leaching out' the sample with excess H_2O. This entails the measurement of small amounts of D_2O in H_2O and various techniques are available for this. Attempts are being made to develop the 'falling drop' method (a density method) to a sufficient degree of accuracy.

Other work being done at the B.R.R.A. is on the crystallisation of polymer cellulose, the feeding of low degree of substance on the structure formation in regenerated cellulose, the molecular arrangement in cellulose, and light scattering of cellulose.

For the last two or three years the association has been developing a spotting agent or cleaning fluid for the removal of oil and dirt stains in cloth. This is now being marketed commercially by Booth and Openshaw Ltd.

A new arrangement announced at the Open Days was a hiring service for MANRA instruments (instruments developed by the association).

Also on view was a high-speed cine unit, capable of taking pictures in the range of 150-5,000 pictures per second.

CHEMICAL EXCHANGE PROCESSES FOR DEUTERIUM PRODUCTION

Precise Optimum Values Still Classified

DESIGN of a dual-temperature chemical exchange process for the production of deuterium was discussed by Mr. C. J. Lyons, Chemical Engineering Division, A.E.R.A., Harwell,* at the joint symposium on 'Instrumentation and computation in process development and plant design', held under the aegis of the British Conference on Automation and Computation, and sponsored by the Institution of Chemical Engineers, the Society of Instrument Technology and the British Computer Society. Venue of the symposium was Church House, Westminster, London S.W.1, on 11, 12 and 13 May.

In design of plant for operation of cascade-type processes, optimisation calculators are usually carried out in order to determine the most favourable operating conditions. Dimensions of the plant and its energy requirements will be based on these. Manual calculations are laborious and time-consuming. Errors easily occur, and checking is as laborious and time-consuming as the original computations. A computing machine can therefore be effective in reducing the time and removing sources of human error.

Lyons's paper dealt with the first stage only of a cascade process in which L_1 represents the large input flow (natural water in this case with its low atom fraction X_1 of deuterium), while P is the small flow of product, highly enriched water with atom fraction X_p of deuterium. Separation is made possible by an equilibrium process taking place between the input water stream, L_1 and rejected stream $(L_1 - P)$ of depleted concentration $Y_1 = kX_1$, k being less than unity.

Taking a deuterium balance around the general or *n*th stage, and the last Nth stage:

$$L_n X_n - (L_n - P)\,kX_n = PX_p$$

This equation is simplified to

$$L_n = \frac{PX_p}{X_n(1-k)}$$

Since $L_n \gg P$ in the early stages and indicates that for a given separation process the rate of flow to the *n*th stage varies inversely as the deuterium concentration.

In the ideal cascade, with an infinite number of stages, the flow rate was reduced continuously, stated Lyons, towards the enriched end, the tower cross-section diminishing in proportion. Obvious advantages of the ideal cascade were reduction of total volume and progressive reduction of the hold-up of increasingly enriched water. In principle, however, the process could be carried out in a single stage without increase in heat requirements. Practical requirements led to the stepped cascade in which each stage for the $H_2O - H_2S$ exchange process included a hot and a cold tower each of which comprised a large number of transfer units. Beyond a certain position in the cascade there were practical advantages in changing from the exchange process involving H_2S to a different exchange process, in particularly because of the risk of losses from the circulating systems of H_2S highly enriched in deuterium. Thus, either water distillation or electrolysis could be used for the later stages, for while the energy requirements of these processes were impractically high for natural water they were negligible when the atom fraction of deuterium was 0.1 – 0.2.

Precise optimum values were deliberately avoided, said Mr. Lyons, as many aspects were still confidential.

In the process described liquid feed of low deuterium concentration was enriched in deuterium by flowing in a packed or bubble-cap tower at a low temperature counter current to a hydrogen-containing gas. A portion of the enriched liquid leaving this tower was withdrawn as product; the remainder was passed through a similar tower at a higher temperature. There the deuterium was stripped from the liquid by gas re-cycled from the top of the cold tower.

The physical data and mass-transfer equations being known, the equations were programmed for an electronic digital computer so that effects of different process conditions could be compared and an optimal set chosen. Operation of the system could be represented by a diagram of the McCabe-Thiele type. This diagram was then elaborated to include the effects of humidity and solubility. Afterwards it was necessary to establish compositions and flow rates for the various gas and liquid streams leaving and entering the exchange towers, the humidifier, the gas saturator and the condenser-cooler. Derivations of these equations were based on mass and material balances—or of the expressions giving the number of theoretical stages required in the hot and cold towers.

Although information on optimum values had to be withheld, Mr. Lyons outlined a fairly obvious procedure for deriving the optimum hot-tower temperature. The first step, he stated, was selec- of the optimum values of G/L (G = circulating hydrogen sulphide stream and L = molar feed rate of water based on the minimum number of total theoretical stages, for different hot tower temperatures, other parameters being the same. The minimum number of total stages, which could be taken as a measure of the combined height of the hot and cold towers, was plotted against the flow rate of humidified gas in the hot tower and the gas flow rate for the cold tower. These flow rates were taken as proportional to the respective tower cross-sectional areas. A rough estimate was then made of the average capital cost per ton D_2O capacity per year, and capital charges worked out for interest on capital, amortisation, maintenance, materials, and labour.

Capital cost was assumed to be directly proportional to tower volume, the capital charge being multiplied by the factor: (minimum number of total stages) (cold and hot flow rates)
for the selected hot-tower temperature. To this capital charge was added the appropriate cost of steam for humidification. This showed the effect of reduction, by means of heat recovery, of the unit cost of steam for humidification, by an arbitrary factor of 0.6. This reduction in steam costs had of course to carry capital charges on the necessary heat exchange equipment in which large gas throughputs were alternately humidified and dehumidified. The costs might well be decisive in comparisons between the present process and other processes.

In the final design engineering considerations, such as the number of first-stage towers in parallel and their shell diameters and thickness, and the choice of materials to withstand the corrosive action of H_2S became of importance.

* *Now Central Technical Services, Industrial Group, U.K.A.E.A., Risley, Lancs.*

New Worthing Factory Heralds Beecham's Expansion in Antibiotics

THE Beecham Group is to begin work shortly on the construction of a new factory at Worthing, as one of the first steps towards expansion into the field of antibiotics. It will manufacture the group's existing range of fine chemicals and products derived from the work of Beecham Research Laboratories, including the new penicillins likely to be evolved as a result of the recent isolation of the basic penicillin molecule.

Beecham Research Laboratories is to be formed into an operating division of the group. It will incorporate C. L. Bencard Ltd., who market the group's pharmaceutical products advertised to the medical profession. This will bring together in one company the responsibility for research, development, manufacture and marketing of all the group's ethical pharmaceutical products.

General pharmaceutical production, the Bencard allergy clinic, and the administrative headquarters of Beecham Research Laboratories will be grouped in the building now occupied by Macleans at Brentford.

To facilitate these moves, the present responsibilities of Macleans for the marketing of proprietary medicines and toiletries will be divided respectively between Beecham Pharmaceuticals and County Laboratories.

U.K. Chemical Output

Board of Trade index of industrial production, based on a 1954 average of 100, puts the output of the chemical and allied industries in 1958 at 115, the same as 1957. The figure for December was 116, against 111 in December 1957.

New Resin Gives Homogenous Laminate with P.V.C. Sheeting

SPECIALLY developed to bond glass fibre reinforcement intimately to a suitably prepared vinyl sheet to produce a strong, homogenous laminate, is Artrite Resin F.655, marketed by Artrite Resins Ltd., Kingsway Chambers, 44-46 Kingsway, London W.C.2. A simple production technique to accomplish this has also been evolved.

Belonging to the Artrite family of L.U. polyester resins, Artrite Resin F655 is formulated for the wet lay-up moulding technique and is supplied ready for use, requiring only the addition of a hardness just prior to moulding. Natural colour of the material is light-buff and translucent, but it can also be supplied in a range of attractive shades to match the colour of vinyl sheeting.

This resin can be used, the makers state, in conjunction with any of the glass fibre reinforcing materials such as glass cloth rovings or chopped strand mat.

Extensive tests have been completed on Cobex sheeting (BX Plastics Ltd.) and Darvic (Imperial Chemical Industries Ltd.). Other types of p.v.c. sheeting are being evaluated. Most grades of rigid vinyl sheeting based on either p.v.c. or p.v.c. copolymers are believed to be suitable for lamination with Resin F655.

Thin gauge sheet as used in vacuum forming, and heavy gauge sheet as used in chemical plant construction have both been successfully bonded. In practice, the method of forming the sheet dictates the gauge to be used.

Conventional methods of fabrication of the vinyl sheet are used, such as vacuum forming, hot pressing, simple heat forming or cutting and hot welding; choice is usually dependent on the size and shape of the article. Formed sheeting must be adequately supported, it is stated, while the glass fibre reinforcement is being laid up; for heavy gauge sheet a skeleton framework is usually adequate. For thin vacuum-formed shapes a close fitting former from wood or plaster may be required.

In surface preparation of the vinyl sheet, to ensure homogenous lamination, sanding is carried out before and after forming followed by cleaning using a special Artrite solvent or a trichloroethylene vapour bath or acetone washing. After the sheeting dries, Resin F655 and glass fibre is applied.

The procedures involved in moulding the glass fibre reinforced resin on to the vinyl sheeting are stated to be identical to those of moulding reinforced plastics by the 'wet lay-up' technique on to a conventional mould. Setting times are adjusted by the amount of hardener employed, but Resin F655 should not be allowed to start to set until about 30 minutes after it has first been brushed on to the vinyl sheet. This allows for adequate reaction between the resin and the p.v.c.

U.K. Compounders Import Ammonium Sulphate

U.K. FERTILISER importers are said to have paid 45s freight a ton for a cargo from Tampa in a small ship, according to *Monthly Freight Review* for April. Other British importers, seeking freight offers to a deepwater port for 125,000 tons spread over the 1959-60 fertiliser year should, if recent business to the Continent is a guide, manage several shillings a ton less than this.

In manufactured products, nitrogenous fertilisers took the lead, but the U.K. did not share in a heavy export business to the Far East. It is stated that imports of ammonium sulphate from Belgium were called for in April to meet the needs of U.K. compounders.

Reports that Communist China must lift some 350,000 tons of fertiliser by end-May are described as grossly exaggerated, but it is felt that some charterings have been concluded secretly at rates well above recent market norms.

Among April freight quotations are the following: *Phosphates*: Casablanca to Immingham, 13,000 tons at 23s 9d per ton (1s 6d load—5s discharge); *Nitrogen fertilisers*: Ghent to Avonmouth, 2,600 tons at 16s 6d per ton f.i.o.; *pyrites*: Domsjo to Middlesbrough, 1,200 tons at 2s 6d per ton, f.o.b. and c.r.d.

Wimpey's Chemical Plant Contracts in the U.K.

IN THE field of petrochemicals, George Wimpey and Co. Ltd. are continuing with the construction of an ethylene oxide plant for Gemec Ltd., Fawley, work that follows completion of a polythene plant in January 1958 at Grangemouth for the same company. These two projects have an approximate total value of £7 million and "have involved particular problems in mechanical design and installation."

This was stated by Sir Geoffrey Mitchell, chairman, in his review of 1958 to shareholders. Other projects the company is engaged on include ethylene oxide and oxygen plants for the Lummus Co., on behalf of Petrochemicals Ltd., at Carrington, a low-pressure polythene, cumene and phenol plant at Grangemouth for Stone and Webster Engineering on behalf of British Hydrocarbon Chemicals, and construction work at Stanlow for Shell.

During the year, British Oxygen Wimpey maintained steady progress on the special rocket project in Cumberland for the Ministry of Works.

Substantial contracts in progress included site preparation for the new oil refinery under construction by Foster Wheeler for Esso at Milford Haven, and I.C.I.'s new Terylene plant at Wilton.

QVF Introduce Chemical Glassware Erection Service

A new erection service for the chemical industry has been introduced by Q.V.F. Ltd., Fenton, Stoke-on-Trent, and is staffed by technicians who have received specialised training in the erection of the company's products. This is designed to complement the assistance which Q.V.F. offer with design and erection methods.

Mr. J. G. Window, sales director of Q.V.F., says "Our engineers are continuously engaged in the erection of glass plant and pipeline which is used in almost every country of the world. In addition to supplying customers with glass we are naturally interested to see that the material is put into service satisfactorily and is able to function with the highest degree of efficiency. We hope that customers will take full advantage of this new service."

Erection charges are based on an hourly rate plus travelling and accommodation expenses. However, if required, the company will submit an estimate for the service. Breakages occurring during erection, for which Q.V.F. staff are responsible, are made good at the company's expense.

Lack of Demand Leads to Closure of H_2SO_4 Plant

SUBSIDIARY company of William Blythe and Co. has been unable to continue its operation of its sulphuric acid plant at Hapton due to difficult trading conditions. Technical difficulties experienced during construction and in starting up and initially operating the plant, together with lack of demand for acid, had resulted in intermittent operation. As a result appreciable deterioration had occurred in various sections of the plant.

This was stated by Mr. W. Collinson in his annual report to shareholders. It appeared that fairly heavy expenditure would have to be incurred to bring the plant into efficient production and the board are employing a consulting chemical engineer to report independently before reaching a final decision as to the future of the plant. A special provision for depreciation has been set aside over the last few years and when a final decision can be reached it will probably be necessary to provide for further substantial depreciation on the plant.

Index of Chemistry Films

An index of chemistry films compiled by the Royal Institute of Chemistry aims at bringing together in one volume details of all films and filmstrips on chemistry and related topics available in this country. It is designed to cover the requirements of schools, technical colleges, universities and the training departments of industrial firms.

The major sections of the index consist of lists of about 800 films and 200 filmstrips, classified by subjects. These are followed by the addresses of distributors and an alphabetical list of all the titles.

Copies may be obtained from the Education Officer, 30 Russell Square, London W.C.1, 5s post free.

O.C.C.A. POLYMER CONFERENCE—2

Work on Boron Polymers Might Lead to High Temperature Resistance

EVIDENCE of the considerable activity in the investigation of boron chemistry from the viewpoint of applicable polymers, or materials which might be used as beneficial additives in the technical production and uses of known polymeric materials was given by Dr. W. Gerrard, Northern Polytechnic, London, at the Oil and Colour Chemists' Association Conference in Edinburgh.

Emerging from certain spasmodic reference to polymeric material produced in the very small-scale experimental work on boron hydrides and borazole, and particularly from reports of the Upson patents, and the boron imine, there developed, stated Dr. Gerrard, a feeling that organo-boron and related compounds of boron might provide the materials required—namely high-temperature resistant polymers. In his paper, he outlined the systematic study of certain linkages which might be thermally stable and lead to the development of applicable polymers.

Boric Esters

Boric esters from ethylene glycol. By reacting glycol and boric acid or boron trichloride distinct monomeric compounds had been obtained (ethylene boric acid solids, ethylene chloro boronate, ethylene alkyl or aryl borate, triethylene diborate, tetraalkyl (tetraaryl) ethylene diborate or 2-hydroxyethyl ethylene borate solid). Ethylene chloro boronate had "remarkable thermal stability compared with that of non-cyclic compounds". It remained to be seen, said Dr. Gerrard, if non-vicinal glycols would yield high polymers of hydrolytic stability, but the prospect was not bright.

Boric esters from di- and polyhydric phenols. All the products produced-*o*-phenylene chloro boronate, methyl *o*-phenylene borate, *tris-m*-phenylene diborate, etc.—hydrolysed easily.

Boron-nitrogen systems. Synthesis of *scorpion* boronic acids had been attempted and research had been done on substituted borazole. There were a number of odd statements made about B—N—H compounds, and as much larger amounts of starting materials were now more easily prepared, a complete experimental review of these should be made.

Attachment of alkyl or aryl to boron. The lecturer and his colleagues had described satisfactory laboratory procedures for preparation of the dichlorides. Under the conditions used boronic acid gave trimers (e.g. $(PhBo)_3$) which probably had the boroxale structure. These were easily hydrolysed.

Potentialities in the borazole system. Ruigh *et al.* (Wright Air Development Centre, *Technical Report*, 55-26, 1955, 1956, Parts I to IV) had opened up a field of operations having intriguing potentialities and many obvious schemes presented themselves, said Dr. Gerrard. The interaction of *p*-phenylene diamine with *p*-phenylene-bis-boron dichloride ($Cl_2BC_6H_4BCl_2$) had interesting possibilities. He and his colleagues' main approach to the borazole system had involved production and properties of substituted B-trichloroborazoles and some attention had been given to the reaction steps leading to the formation of the required chloroborazole.

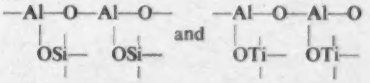
It had been found that the B-trichloro-N-triaryl borazoles, $Ar_3N_3B_3Cl_3$ (where Ar = phenyl, *p*-totyl, *p*-anisyl, *p*-bromophenyl) had good thermal stability, but at higher temperatures underwent further polymerisation, accompanied by elimination of hydrogen chloride. This was interesting, stated Dr. Gerrard, and useful results might emerge from investigation in that part of the project. These borazoles had been alkylated by means of the appropriate Grignard reagent, and it was hoped to get helpful results from a bi-functional Grignard.

Boron-phosphorus polymers. There had been no report on even a near approach to an applicable polymer by Burg *et al.* Dr. Gerrard, however, has approached the production of a phosphorus-boron linkage rather differently, with the intention of avoiding borane-based chemistry. So far results had led on to a P—O—B linkage. The conclusion had been reached that the boron atom became attached to the double-bonded oxygen atom, forming a complex which could be isolated at 23°C, but slowly evolved *n*-butyl chloride and set to a glass when there was still a considerable amount of easily hydrolysable chloride present. When the system was heated slowly to 300°C (6 hours), butyl chloride (1.95 mol.) was evolved, and a red solid residue was produced which at 900°C afforded a white solid believed to be boron phosphate. Hydrogen chloride (0.78 mol.) was also evolved towards the end of the initial heating period.

Dr. Gerrard reported that hydrogen, (as HCl) would probably be evolved from each phosphorus atom when all the butyl groups had been removed. The observed result would be the gradual development of evolution of HCl towards the end of the elimination of butyl chloride. The final result would be a polymer comprising a network of —P—O—B—O— links, giving an easily hydrolysed material, which at 900°C in air, would become a hydrolytically stable form of boron phosphate (a report on this system will be published shortly).

—B—O—P—O—linkages. Interaction of triethyl phosphate and boron trichloride involved primary complexing ($(EtO)_3 P = O. BCl_3$), and a gradual elimination of ethyl chloride, eventually affording an easily hydrolysed solid, almost free from chlorine, containing practically all the phosphorus and boron in the system. On heating to about 1,000°C a fine specimen of boron phosphate was produced. It was hoped that by having certain hydrocarbon groups attached to phosphorus and boron ($R—P(O)(OR')_2$, $R''BCl_2$) polymers which had useful properties might be obtained.

Other systems. It was stated that interesting results could be expected from the use of reagents, having certain hydrocarbon or substituted hydrocarbon groups directly attached to phosphorus and silicon ($RSiCl_3$, $R'P(O)(OR'')_2$). It was obvious that production of linkages such as



would already be under investigation.

New Types of Macromolecule May Be Possible with Organophosphorus Polymers

PHOSPHORUS-containing polymers fall into two main classes: those in which phosphorus forms part of a side group attached to a polymer chain of orthodox character and those in which the main chain of the polymer itself contains phosphorus. Discussing these, Dr. A. F. Childs and Dr. H. Coates (Albright and Wilson (Mfg.) Ltd.) stated that the former had been the most extensively studied up to the present. The latter class, however, offered the possibility of new types of macromolecule with properties differing fundamentally from those of existing polymers. Representative examples of both classes of polymer were discussed.

Addition polymers. More than 50 unsaturated phosphorus-containing monomers had been prepared. Both allyl and vinyl compounds were represented; broadly it might be said that vinyl phosphorus compounds were rather less readily polymerised and allyl phosphorus compounds on occasions rather more so than better known monomers of similar type. Most of the allyl and vinyl esters formed copolymers with other unsaturated monomers, those with vinyl acetate or methyl methacrylate being of particular interest.

A polymer of diallyl phenyl phosphonate was transparent, had a high distortion point (216°F), a refractive index similar to glass (n 28/D 1.573) and low flammability. Triallyl phosphate, which

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* *First part of our report of the Biennial Conference of O.C.C.A. held at Edinburgh from 5 to 9 May appeared in 'Chemical Age' last week, p. 781*

can be prepared on a commercial scale, had an interesting application in the flameproofing of cloth. Emulsion polymerisation of this substance with halomethanes, particularly bromoform, gave a stable suspension of a low molecular weight adduct, suitable for application to textiles, and capable of further cross-linking to give a completely insoluble high polymer. A partly hydrolysed polytriallyl phosphate had ion-exchange properties.

Condensation polymers. (1) *Polyesters.* The best known were probably I.C.I.'s Phoryl Resins. Because they showed good adhesion to a variety of materials, including metals, it had been suggested that they might be possible components of metal priming paints, although this use might be restricted by their somewhat brittle character.

Patents on Flame-resistant Polyester Resins

Several patents had described the production of flame-resistant polyester resins, by reacting conventional unsaturated alkyl resins of the polyalkylene maleate type with trialkyl phosphates or dialkyl phosphites. The latter compounds trans-esterified readily in the presence of sodium alkoxide as a catalyst and polyalkylene phosphites, formed by the reaction of glycols with the lower dialkyl phosphites had been reported. One application of this reaction—by replacing some of the dimethyl terephthalate used in the formation of polyethylene terephthalate with dimethyl phosphite, the product becomes more hydrophylic, until with as little as 5 molecules per cent of dimethyl phosphate the resulting polymer can be plasticised by water.

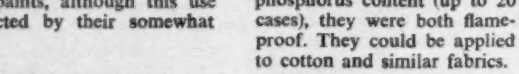
(2) *Polyamides and polyurethanes.* There had been little activity in the field of phosphorus polyamides, Childs and Coates reported.

(3) *Epoxy resins.* Shokal (U.S. Patent 2,732,367, 1956) had observed that polyepoxides, such as low molecular weight glycidyl ether of diphenylopropane, would react with phosphoric acids to give products which contained chemically-bound phosphorus, but were still soluble and fusible. The epoxy phosphoric resins thus obtained could be further cross-linked by normal methods giving flexible flame-resistant materials suitable as potting or coating resins. The patent claims were very interesting, considered Childs and Coates, and deserved further study.

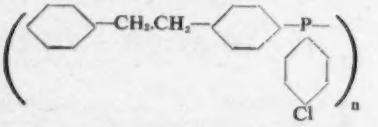
(4) *Phenol-formaldehyde and amide formaldehyde resins.* A recent patent (C.A. Redfarn, British Patent 807,851) had shown that by adjusting the proportions of acid chloride to dihydric phenol, low molecular weight polyesters can be obtained which contained free hydroxyl groups, and could be thermoset to hard bodies by further cross-linking with formaldehyde donors, such as hexamethylenetetramine. The resulting resins could be regarded as phenol formaldehyde resins cross-linked by phosphorus polyester groups and they showed an improved thermal stability over orthodox phenol-formaldehyde resins. Some of the more promising of Redfarn's resins were based on the phosphonitritic chlorides $((PNCl_2)_n)$ which were now commercially available.

Tetrakis (hydroxymethyl) phosphonium chloride (T.H.P.C. $[(HOCH_2)_4P]^+Cl^-$) with phenols gave polymers ranging from viscous liquids to infusible solids. T.H.P.C. and its derivatives formed polymers with amides, such as urea, melamine and methylolated melamines, dicyandiamide and similar bodies which Childs and Coates considered of greater technical interest. These ranged from water-soluble precondensates of low molecular weight to highly insoluble resins. T.H.P.C.—urea polymers closely resembled urea-formaldehyde resins it was reported except by virtue of their phosphorus content (up to 20% in some cases), they were both flame- and glow-proof. They could be applied as finishes to cotton and similar fabrics.

New types of polymer. One obvious approach, in view of the stability of the P—C link, was to synthesise polymers of the structure:



This had proved difficult to do. Korshak (*Izvest Akad. Nauk. S.S.S.R. (Otdel. Khim),* 1958—618) had obtained polymers by heating *p*-chlorophenyl phosphonous dichloride with 1.2-diphenylethylene. No details of the stability of these polymers were available, but Childs and Coates suggested that if they had the structure suggested they would be likely to break up on heating to give smaller unsaturated units.



Attention had been paid recently to polymers based on the phosphonitrilic skeleton, —P = N—

$$PCl_5 + NH_4Cl \longrightarrow (-N=PCl_2)_n + 4HCl$$

They formed an extensive series of cyclic polymers, of which the lowest (n = 3) trimeric phosphonitrilic chloride had a formal resemblance to benzene.

Somewhat more reactive than the cyclic polymers were the less well-defined series of linear polymers of the general formula $[(PNCl_2)_n\ PCl_4]$.

Of other classes of polymer with an essentially inorganic skeleton, Andrianov (*J. Polymer Sci.,* 1958, **30,** 1513) had exampled complex poly-trialkylsiloxy-aluminophosphates. Polyborophosphates had also been reported but were deemed too hydrolytically unstable for commercial application. The phosphinoborines showed far greater promise.

Dr. Grassie of Glasgow Discusses Deterioration of Synthetic Polymers

DEGRADATION reactions trouble the manufacturer in two phases of the life of polymers—during fabrication and in subsequent processes. Three agencies—heat, light and oxygen—are usually involved either singly or in combination. Dr. N. Grassie, Chemistry Department, Glasgow University, discussing the chemistry of the deterioration of synthetic polymers, gave a general picture of the sort of processes which occur.

With very high energy radiation, X-rays, α-rays, or neutrons, plastics such as polystyrene are rapidly insolubilised. When radicals formed are not stabilised as they are in rubber and polystyrene as in polymethyl methacrylate or polythene, then decomposition effectively competes with cross-linking. Cross-linking in pure rubber results in a tougher material that could be used over a wider range of temperature and is usually achieved by vulcanisation, i.e. by treatment with sulphur. When it is not desired to have sulphur present, other methods of vulcanisation have been used. Radiation cross-linking, says Dr. Grassie, would appear to offer the ideal solution, since no additives are required and the extent of vulcanisation can be closely controlled.

Oxygen and light is a potent combination in attacking polymers. Light produces radicals which react with oxygen to form peroxide radicals, which abstract a hydrogen atom elsewhere in the system to reform the original radical. It is not the hydro peroxidation process itself which is responsible for the gross physical changes in oxidised polymers. It does appear, considers Grassie that the principal primary mode of decomposition is rupture of the oxygen—oxygen bond. The oxyradical, RO, is very much more liable to undergo scission reactions than the parent radical, R. Therefore, while light alone results in hardening of rubbers, admission of oxygen can, under certain circumstances, cause softening.

Chemically, ozone-cracking clearly consists of direct attack at double bonds by ozone, followed by chain scission, but it is very much more difficult to account for the highly concentrated nature of attack at relatively few points.

An explanation of the extreme susceptibility of stretched rubbers may be found in the reasonable assumption that chain scission into radicals is part of the ozonisation process. In relaxed rubber these would combine, while in rubber under stress there will be permanent separation.

Colour in organic compound is usually associated with unsaturated groupings such as C = O, C = C, C = N, etc., Dr. Grassie notes. The reactions involved illustrate rather well "the sort of *zipper* mechanisms which are possible only because of the special structural nature of polymer molecules".

In p.v.c. and p.v.a. the reaction

$$-CH_2-CHCl \rightarrow CH = CH + HCl$$

on heating the plastics material is responsible for coloration. It is a classical ester decomposition reaction into acid and olefin but once one acid molecule is lost from the saturated chain, and this requires a relatively large amount of energy, the double bond so formed activ-

ates the α-hydrogen atoms.*
—CH = CH—CH_2*—CHCl—CH_2—CH-Cl and the adjacent acid molecule is liberated with absorption of very much less energy. "So the reaction tends to zip along the chain, conjugated structures are rapidly built up and colour develops which moves through the spectrum from yellow to red."

While ultimate stability of a commercial plastic based on a polychloro resin depends upon choice of filler and plasticiser, the most important Grassie says is the efficiency of the stabiliser. Deteriorative weathering process is illustrated by esters of maleic and fumaric acid which undergo Diels-Alder reaction. Antioxidants like di-*tert*-butyl-*p*-cresol are beneficial in preventing formation of chlorine-labilising structures, such as carbonyl groups. Also a high degre of stabilisation towards light degradation can often be achieved by completely opaque substances like carbon black or titanium pigments to selective ultra-violet absorbers, like phenyl or heavy metal salicylates. Finally incorporation of substances which react with hydrogen chloride have considerable stabilising power although their affinity for hydrogen chloride must not be such as to strip the chloride from the polymer chains.

Discoloration is troublesome in the nitrile polymers. Colour here is due to conjugation produced by the linking up of adjacent nitrile groups, which are already fixed in positions ideal for reaction.

Invariably, notes Dr. Grassie, polymers are less stable than one would predict and sometimes undergo entirely different types of reactions from model compounds. The reason for this is that the reactions which occur are often chain processes intiated at labile abnormalities in the polymer structure. These are usually present in minute concentration but their effect is magnified by the long chains of reaction which they trigger off. Thus while hexadecane is stable at 400°C, polythene shows signs of decomposition at 290°C. This is due to the highly branched nature of polythene and the low concentration of carbonyl groups, as a result of oxidation or copolymerisation with carbon-monoxide present as impurity in the monomer. The carbonyl group, being the least thermally stable part of the molecule breaks down to radicals, which attack the tertiary hydrogen atoms at the branches, resulting in molecular scission.

Dr. Grassie reports that most of the coloration in polymethacrylonitrile obtained from monomer as normally prepared is in fact caused by the presence of traces of methacrylic acid polymerised into chains. A wide variety of proton-donating substances including acids, phenols, imides, and some ketones, aldehydes and alcohols have the same effect.

Coloration in polyacrylonitrile is initiated in precisely the same way, but the residual coloration remains even when purification and polymerisation precautions are observed. Dr. Grassie says that this is caused by the fact that the tertiary hydrogen atoms in polyacrylonitrile are themselves reactive enough to initiate the reaction.

Thermal decomposition of polymethacrylates illustrates a completely different type of reaction. During polymerisation of this polymer, the growing radicals are ultimately destroyed by reaction in pairs so that half of the chains have saturated and the other half unsaturated terminal groups. By modifying the polymerisation system so as to control the structures of the chain ends, stability of the material can be influenced.

Uses of Elastomeric Polysulphides as Coatings and Sealants

DETAILS relating to the development and utilisation of the elastomeric polysulphides were dealt with by Mr. W. H. Stevens, of Bromley, Kent. Although the first developments in this field were noted in the 1920's both in Europe and the U.S. it was as a result of research and development during the last 10 years or so that interest and knowledge grew in application of these liquid polymers. Major fields of applications lie in coatings and sealants and some aspects of this experience were discussed by Mr. Stevens.

Straight polysulphide coatings. Liquid polysulphides could be used in the formulation of coatings wherein the polysulphide forms the main basis of the medium and also in coatings where the polysulphide is used as a modifying agent for synthetic resin.

The first class consisted of liquid polysulphide, pigments, filters, etc., ground together on a triple-roll mill and thinned with solvent. Carbon black had proved to be the best pigment where the colour was acceptable, because of the toughening effect which the pigment had on the finished film.

Numerous variations of the formula—

Polysulphide paint LP2 black	*Parts by weight*
LP2	100
SRF black	30
Stearic acid	1
Phenolic resin	5
Methyl ethyl ketone	40
Toluol	41
CS curing agent	15

CS Curing Agent	*Parts by weight*
Lead peroxide	50
Dibutyl phthalate	45
Stearic acid	5

in order to alter the properties of the composition and coating were possible; for instance, chlorinated solvents such as trichloroethylene could be used when a non-inflammable paint was required, and by increasing the stearic acid to 3 parts the set time was doubled. Adding 4 parts of ultra fine silica to the above formulation increased film thickness from 8 ml. to 18 ml. per spray coat.

The use of lead peroxide as curing agent was precluded in the case of white or light-coloured polysulphide paints because of its colour. Antimony trioxide, however, might be found suitable, or alternatively, organic peroxides. These tend to be slower curing than lead peroxide and less effective in producing a tack-free product, but traces of lead driers enhance their activity. Cyclohexanone peroxide in the presence of 0.1% of lead as lead naphthenate, gave a very rapid cure. Tertiary butyl hydroperoxide which did not react on its own in less than 2-3 days even then produced a tacky end product, with lead naphthenate present, it gave a useful set time of about 16 hours at 70°F and a firm product.

Polysulphide resin coatings. Pride of place had to be given to polysulphide/epoxy resin blends. A hardening agent, usually an amine was necessary. Aromatic amines could be pre-blended to give stable compositions with liquid polysulphides and in the case of aliphatic amines pre-blending could also be practised, but the product was not package stable. Dilution of the amine by pre-mixing with liquid polysulphide reduced the degree of toxicity associated with amines.

Blends with other resins. A potentially valuable application of sulphides in connection with other resin media, Mr. Stevens stated, was in blends with polyesters and with vinyl esters.

A blend such as the last mentioned—polysulphide and polyvinylidene chloride—was used in the internal coating of concrete storage tanks for petroleum fuels. In a reverse-phase composition, polysulphide aqueous dispersion was added to a solution of vinyl copolymer resin in ketone solvent; the polysulphide and resin became blended together and the water separated and was discarded.

Polysulphide sealants. Modern polysulphide sealants were based on the liquid polysulphides which were compounded and then catalysed to the solid stated *in situ*. These sealants had provided, Mr. Stevens observed, a complete answer to the extreme problem of sealing curtain wall structures. U.S. manufacturers of polysulphide building sealants were at present engaged in drawing up a specification to cover the performance and testing of these materials. It was expected that this specification, when finalised, would cover two classes of sealing compound, i.e. the self-levelling (within a temperature range of 40°-120°F) and the non-sag (for application in vertical seams at 40°-100°F).

Formulation of Polysulphide sealants. As in the case of polysulphide coatings, it was possible also with sealants to incorporate all sorts of pigments, extenders and plasticisers. A useful ingredient for improving adhesion and water resistance was tar providing the black colour was permissible.

When phenolic resins can be used in the sealant composition, adhesion is stated to be greatly improved. These resins were not permissible in compositions for use on white stone because a pink discoloration could be produced in time. In such cases, and even in conjunction with phenolic resins, improved results could be obtained by including vinyl acetate resin in the composition.

Acknowledgment was made by Mr. Stevens to Thiokol Chemical Corporation, Trenton, New Jersey, U.S., for data included in his paper.

Other papers presented at the O.C.C.A. conference will be reported next week.

Overseas News

MANUFACTURING LICENCES GRANTED TO INDIAN CHEMICAL PRODUCERS

A LICENCE for the construction of a caustic soda plant to produce 7,000 tons a year on a single shift basis has been granted by the Government of India to Mettur Chemicals and Industrial Corp., Mettur Dam, Salem District. Otul Products Ltd., Surat, Bombay State, have been granted licences to produce caustic soda (150 tons/month); potassium hydroxide (150 tons/month) chlorine (240 tons/month); hydrochloric acid, 100% (200 tons/month).

Other licences granted include: Excel Industries, Bombay, cuprous oxide (60 tons/year), cupric chloride and cuprous chloride (50 tons of each per month), ferric chloride (250 tons/month), mercury salts (25 tons/month); Beco Chemicals Calcutta 13, ferrous sulphate (500 tons/month); Tata Chemicals Ltd., Fort Bombay, hydrochloric acid (495 tons/month); Hoechst Fedco Pharma, Bombay 1, p.v.a. in dispersion (200 tons/month); Elephant Oil Mills, Bombay, alkyd resins (10.5 tons/month); Alta Laboratories, Bombay, pentachlorophenol (120 tons/year); Sardesai Bros., Bilimora, pilot plants for pentachlorophenols and sodium pentachlorophates. Indian Oxygen Ltd., Calcutta 27, expansion of capacity for dissolved acetylene from 0.36 million cu. ft. per month to 0.54 million cu. ft. Colour-Chem Ltd., Bombay, phthalocyanine blue (100 tons/year).

Italy to Take More Dollar Imports

The Italian Government is to liberalise imports from the dollar area. With a large-scale programme of plant erection and development to take place, the liberalised quotas for capital goods in particular are to be increased. The quotas in the case of chemical equipment will rise to 80% of all imports and that for precision instruments for the chemical and other industries to 70%.

Dimethyl Sulphoxide as Solvent for Acrylic Fibre

Dimethyl sulphoxide (DMSO), marketed by Stephan Chemical Co. U.S. is to be used as spinning solvent in production of a new type of acrylic fibre by Toyo Rayon Co. Some 1 million lb./yr. is being produced by this company and it is believed that there might be a market for as much as 10 million lb./yr. With greatly increased volume production the current price of 34 cents/lb. is expected to be reduced to 25 cents/lb.

Use of DMSO as acrylic fibre solvent is not only this solvent's first large-scale commercial application. It is suggested that it is its best hope for increased growth. Several other Japanese manufacturers who plan to produce synthetic fibres are expected to employ DMSO. A Japanese patent issued in the U.S. last year covers use of DMSO as a solvent for an acrylic fibre, the description of which resembles Zefran, produced by Dow Chemicals. According to the patent polyacrylonitrile is dissolved in DMSO and the fibre spun into a dilute DMSO bath.

Reports from the U.S. (*Chem. Engng.*, 1959, **66**, No. 8, 73) indicate that E. I. Du Pont de Nemours Co. have been examining the possibilities of using DMSO instead of dimethyl formamide in production or Orlon. Interest has also been expressed by Chemstrand Corporation.

Peruvian Fertiliser Factory Starting Up

An annual output of 20,000 metric tons of anhydrous ammonia, 54,000 tons of nitric acid, 35,000 tons of ammonium nitrate and 7,000 tons of ammonium sulphate is expected from the new fertiliser factory built by Fertilizantes Sineticos S.A. in Callao, Peru, which is going into production.

The plant was designed and equipped by the Italian concern Montecatini. Half the capital of $10 million was contributed by Peruvian investors and half advanced by an Italian bank on a long-term basis.

U.S. Firm to Build Petrochemical Plant in Argentina

The Government of Argentina has granted a request of the Houston petrochemical and chemical producer, Texas Butadiene and Chemical International Ltd., to be allowed to erect plant near the main Argentine oilfields in the Comodoro-Rivadavia area for the production of butadiene, styrene and gas carbon black. The American company is to invest up to $67 million in the scheme.

Three East European Synthetic Rubber Plants

The Polish synthetic rubber plant at Oswiecim will not, as was hoped, reach an initial annual capacity of 6,000 tonnes but only 1,500 tonnes owing to hold-ups in the construction programme. The future targets for the completed and later extended plant stand at present unchanged at 20,000 tonnes by next year and as much as 70,000 tonnes by 1970. Other news from behind the Iron Curtain is that the Czech authorities have now announced that the first stage of Czechoslovakia's new synthetic rubber plant at Kralup will be finished by 1963. It is hoped that the new plant will supply half of the country's demand and cut imports, which for all types of rubber equalled 53,000 tonnes annually by the most recent statistics. A synthetic rubber plant with an annual capacity of 45,000 tonnes and a p.v.c. installation with a 33,000-tonne capacity annually are to be added to the giant Borzesti chemical Kombinat in Rumania.

Colgate-Palmolive Enter Pharmaceutical Industry

The Colgate-Palmolive Co., New York, are to enter the proprietary and ethical drug fields, with the establishment of a new division within the company's organisation, to be known as the Drug Division.

U.S. Polypropylene Plant in Operation Soon

A polypropylene plant with an annual capacity of 90,000 short tons is to come into operation in the next few weeks at the main plant of the United States Avisun Corporation.

Frankfurt Pyrolysis Plant

The Frankfurt-on-Main company Farbwerke Hoechst are constructing a high-temperature pyrolysis plant on their main production site. The plant will obtain crack gases from oil derivatives at temperatures of 2,000°C, the gases then being processed to acetylene and ethylene.

Synthetic Fibre Developments in U.S.

Vinylon, polyvinyl alcohol fibre imported from Japan at 85 cents/lb. f.o.b. is being marketed in the United States by Air Reduction Inc. Airco were licensed to make Vinylon by Kurashiki Rayon; the company have made no decision as yet with regard to plant facilities.

Rogosin Industries (New York) Inc., have announced plans to make 2.5 million lb. of nylon-6 and 2.5 million lb. of polyester fibre a year at Ashdod, Israel. Nylon production is to start 1 January 1960.

Italian-built Plant For Yugoslavia

The Italian concerns Montecatini and Ansaldo are to build a plant for the production of nitrogen fertilisers at Lukavac, Yugoslavia.

Utilising the residue gas of the Boris Kidric cokery, the plant will be large enough to produce 100 tons of ammonia, 335 tons of nitric acid (53%), and 375 tons of nitro-calcareous mineral a day. Later on this capacity will be doubled.

The plant will cost about 9,000 million lire (about £5 million), will take about three years to build and to start production.

Israeli Help with Brazilian Phosphate Plant

Fertilisers and Chemicals Ltd., Haifa, are to supply technical knowledge and experts for the construction of dicalcium phosphate plant in Brazil for Nitro Quimica Brasileira of Rio de Janeiro.

ANALYSIS OF TAR ACIDS—I

H. G. Willcock Discusses Useful Chemical and Physical Methods

BECAUSE of the importance of phenols—tar acids—to the chemical industry a considerable amount of work has been carried out on detection, separation and analysis of this class of compound. In his paper, 'The analysis of tar acids,' presented at a recent meeting of the Midlands Section of the Society for Analytical Chemistry in Nottingham, Mr. H. G. Willcock (Midland Tar Distillers Ltd.) surveyed the more useful chemical and physical methods available for analysis of phenols generally. Techniques applicable to specific problems, primarily isolation of the phenolic portion of a mixture in a form which could be subjected to one of a limited number of analytical methods, had been adequately reviewed from time to time. (1). Neither chemical nor physical properties of phenolic isomers varied greatly and there were, therefore, few methods specific for a particular phenol. Most methods determined phenols as a class rather than individuals, said Mr. Willcock.

Hydroxyl Group

Among general methods of chemical analysis, determination of hydroxyl group, e.g., by esterification, active hydrogen determination or non-aqueous titration, was not specific for individual phenols in their mixtures. It could only apply to non-hydroxylic solutions, for obvious reasons, and gave, therefore, only limited information. Acetylation was satisfactory for phenols other than those having large groups on the 2- and 6-positions. Phthalation was, however, slow and unsatisfactory.

Active hydrogen could be readily determined by the Zerewitinoff method using a Grignard reagent or with lithium aluminium hydride. Such methods were useful for determination of equivalent weights of some sterically hindered phenols.

Apparent acidities of phenols were enhanced in non-aqueous solutions of strong bases and phenols could be titrated either potentiometrically or visually with non-aqueous alkali solutions. Dimethyl formamide was a suitable solvent for stronger phenols but diethylamine was to be preferred for weaker alkyl phenols. Titrants included sodium amino ethoxide, sodium and potassium ethoxide and tetrabutyl ammonium hydroxide in the amine or alcoholic solution. Suitable indicators were o-nitroaniline and azo violet.

In potentiometric titrations, antimony and polarised platinum electrodes had been found satisfactory. The normal glass electrode was insensitive in presence of sodium ions but, otherwise, quite satisfactory in conjunction with a sleeved calomel electrode (2), particularly if the potassium chloride solution in the latter was made up in methanol.

More recently, tetrabutyl ammonium hydroxide in isopropanol (3) or benzene-methanol (4) had been used for potentiometric titration of phenols. Mr. Willcock had used these methods for determination of phenols in hydrocarbon oils and in cyclohexane eluates from a chromatographic column. Cetyl trimethyl ammonium hydroxide had proved just as satisfactory as had tetrabutyl ammonium hydroxide.

Lithium aluminium hydride in tetrahydrofuran might also be used to determine phenols in oils. Excess reagent was added and the excess back-titrated potentiometrically with isopropanol in benzene.

In theory, bromination of phenols offered a relatively simple and rapid method of analysis and it had, in fact, been widely used. The method was essentially that of Koppeschaar in which phenols, in aqueous solution, were brominated by bromine liberated from acidified bromide-bromate solution. Excess bromine was determined in a normal manner. The main disadvantage (5, 6), confirmed by Mr. Willcock and his colleagues, was that phenols took up non-stoichiometric quantities of bromine. This abnormality was influenced particularly by the excess of bromine and temperature and to a lesser extent by time and acid concentration. Iodination had been used to determine certain *o*-substituted phenols but it was open to similar criticism to that of bromination.

Colorimetric Reagents

Most colorimetric reagents used were neither specific for individual phenols nor completely reliable for determination of total phenols in solution under all circumstances. Such reagents were, however, widely used for determination of low concentrations of phenols in aqueous solution.

One colorimetric method (7) depended on reduction of a complex molybdenum or tungsten compound, e.g. phosphomolybdic or phosphotungstic acid, to give a blue colloidal solution of the metal oxides. It was obviously non-specific and the colour faded on standing. Concentrations of phenol up to 0.6% might be used.

Another colorimetric determination was based on the well-known coupling of phenols, possessing free *o*- and *p*-positions, with diazonium salts. Diazotised *p*-nitroaniline (8) and diazotised sulphanilic acid (9) had been commonly used. Care had to be taken in rate of addition of alkali to the aqueous phenol and diazonium salt solution since coupling occurred at appreciable speed over a fairly narrow pH range. Buffering of the solution was recommended. Furthermore, coupling, while essentially in the *p*-position, could also take place in the *o*-position, and hydrolysis of the diazonium salt itself to a phenolic compound was possible.

More recently, a stabilised diazonium compound, the naphthalene-2-sulphonic acid salt of 2-chloro-4-nitro-diazobenzene (N.N.C.D.), had been used, thereby avoiding the necessity of separate preparation of diazonium salt solutions. Although stable in the solid state, in aqueous solution it decomposed as readily as the normally prepared salts. Similar precautions had to be taken in its use to those described earlier.

Quinonoid Salt

Phenols reacted with nitrous acid, supplied in the form of sodium nitrite and acid, to give nitrosophenols, which rearranged in presence of alcoholic ammonia to a coloured quinonoid salt. This reaction formed the basis of a colorimetric method of analysis (10), the reaction being carried out in a buffered solution of sodium acetate. It had the advantage of having some specificity in that colours given by some phenols had adsorption maxima at different wavelengths and this feature might be used to analyse binary mixtures of *m*- and *p*-cresols and of 2 : 4- and 2 : 5-xylenols to an accuracy of ±2%. Care had to be exercised in the neutralisation stage to avoid any appreciable temperature rise. Disadvantages were that at least 12 hours were required for full colour development and that aromatic amines interfered.

Millon's reagent, a solution of mercury in nitric acid, gave a colour with phenols believed to be due to condensation products of nitrosophenols. Chapin had used the reagent to determine phenol in cresylic acid by virtue of the fact that the colour developed by phenol was bleached by formaldehyde. This was essentially the method described in the S.T.P.T.C. Handbook (11). The reagent was unstable and it needed to be prepared daily. Both time and temperature of reaction affected colour intensities.

Phenols underwent oxidative coupling with 1 : 4-amino-phenols or aromatic 1 : 4-diamines to give coloured indophenols or indoanilines. Such reactions were extremely sensitive and might be made more so by concentration of the dyestuff by extraction with a solvent such as *n*-butanol. Phenol concentrations of less than 1 p.p.m. might be determined. The couplings required a pH greater than 7 and usually in the range 9-10, maintained by a borate buffer.

4-Amino-dimethyl-aniline (12), with sodium hypochlorite as oxidising agent, and 4-amino-2 : 6-dibromophenol (13), a more stable reagent, had both found

application. The best known method was, however, that in which the oxidation stage was avoided by using 2 : 6-dibromoquinone chlorimide (**14**). This reagent had been very widely used, although colour development took 18 to 24 hours.

Obviously an open *p*-position was required and therein lay one disadvantage in determination of mixed phenols. Time, temperature and pH (**15**) were important factors affecting this method.

Allied to the indophenol-type reaction was that of phenols with 4-aminoantipyrin (**16**). This reagent reacted with phenols under alkaline conditions in presence of an oxidising agent. Again, a free *p*-position was essential, although some groups, e.g. Cl–, –COOH, $-SO_3H$, –OH and –OMe, were displaced if present in that position. *P*-Alkyl-phenols gave, however, virtually no colour and some use might be made of this in analysis of simple mixtures of *m*- and *p*-isomers.

Advantages of the method were that the colour given by all phenols had the same spectral characteristics, although to differing intensities, the reagent was stable and colour development rapid, and control of pH, an important factor, was readily resolved.

Certain other chemical treatments had been evolved for individual cases and one widely used for determination of *m*-cresol in commercial *m*-, *p*-cresol mixtures (**17**), was based on nitration of such mixtures, followed by separation and weighing of the solid nitro-*m*-cresol. Again, this was a non-stoichiometric method and it had to be carried out under carefully stipulated conditions so that a correction factor might be applied. A second illustration was determination of catechol by precipitation of its lead complex or more recently with ethyl ammonium molybdate (**18**).

Limitations

Owing to the limitations of chemical methods of analysis, more and more attention was being directed to physical and physico-chemical techniques.

Obviously, since distillation was used commercially to achieve some separation of crude tar acids, it could be applied to a limited extent to analysis of such acids. The limitations were severe since many phenols had similar vapour pressures, particularly as the degree of alkyl substitution increased. It could be useful in achieving some simplification of phenol mixtures prior to their more complete analysis by other methods.

Many phenols were crystalline solids at normal temperatures and, if present to sufficient extent in their mixtures with other phenols might be determined by measurement of the crystallising point of the mixture. This property was related to a previously prepared calibration graph. Such methods were used for phenol, *o*-cresol, 2 : 5- and 3 : 5-xylenols.

A number of phenols formed higher melting complexes with a variety of compounds, and crystallising point of tar acid mixtures, in which such complexes had been formed, might be related to percentage of the phenol forming the complex. Methods for determination of the major constituents of phenolic mixtures by such techniques had recently been proposed (**19**). Unfortunately, in many cases, concentration of the phenol was insufficient to apply these techniques without boosting the sample by addition of a known quantity of the pure component. This presupposed an adequate supply of component of sufficient purity and also introduced inaccuracies since the added component must often greatly exceed the amount present in the mixture.

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(*To be concluded*)

New U.S. Rapid Analysis Method Determines Organically Bound Chlorine

DETERMINATION of chlorine in organic compounds economically and rapidly is reported by H. Beckman, Texas Agricultural Experiment Station, U.S. The new technique is stated to facilitate analysis of chlorine in pesticides and can be used to determine the other halogens as well.

An accuracy seldom exceeding 3% error is claimed—in tests of chlorine recovery from insecticides the error has not exceeded 2%. Time taken to complete an analysis is 30 minutes and much less manipulation is required compared with other tests. In fact, several analyses may be run concurrently by one person. Only one piece of apparatus—a beaker—is required throughout the analytical procedure.

The test is carried out as follows: A sample containing about 1 milli-equivalent of chlorine is weighed into the beaker. Excess ammonia is then added to dissolve the sample, together with a little ethyl ether. Two or three small pieces of metallic sodium (0.5 gm.) are added to reduce the chlorine to the chloride ion. Reduction is complete in about two minutes and is indicated by the appearance of a blue colour. Ammonia and the ether are evaporated, and excess sodium combines with moisture in the air to form sodium hydroxide. The blue colour then disappears. Neutralisation of the solution is then effected by nitric acid, and the chloride is titrated with silver nitrate. The end point can be determined either amperometrically, potentiometrically, or visually, depending on the accuracy required.

Sulphur present in the sample can be removed by oxidation to sulphate with hydroperoxide and heat before the neutralisation procedure.

Halogens in polymers such as polyvinyl chloride, polyvinyl chloride and polytetrafluoroethylene (p.t.f.e.) can also be determined. In the case of p.t.f.e., fluorine is determined gravimetrically as calcium fluoride after defluorination.

Need for National Certificates in Instrument Technology—S.I.T. President

THERE is an urgent need for ordinary and higher national certificate courses in instrument technology, Mr. J. F. Coales stated in his presidential address to the Society of Instrument Technology in London on 6 May.

He said he believed the Ministry of Education was considering setting up an approving committee for such courses.

Instrument technology included the fundamentals of chemical, electrical and mechanical engineering as well as applied physics, and it was considered that the certificate should be used as part of the qualification for graduateship of any of these institutions. The committee should include representatives of the Institutions of Chemical, Electrical and Mechanical Engineering, the Institute of Physics and the Society of Instrument Technology.

The Society of Instrument Technology proposed to hold a two-day conference in September to discuss proposed syllabuses.

Mr. Coales said a large part of British industry needed to have 10% of its employees fully qualified technologists as soon as possible. Of the 12,000,000 persons employed in industries dependent on technological advance at least 5% of the fully employed, or 500,000, needed to be qualified technologists now.

At least 500 control engineers and 3,000 instrument technicians needed to be turned out each year.

Iraqi Door "Not Totally Closed" to I.C.I.

IRAQ has not "totally closed the door" to Imperial Chemical Industries Ltd., which has been blacklisted by the Arab League Boycott Bureau, Dr. Ibrahim Kubbah, Economics Minister stated on Tuesday.

I.C.I. were blacklisted for alleged dealings with Israel, which is under economic blockade by the Arab countries.

Dr. Kubbah said it was up to I.C.I. to present "convincing and legal documents" for reconsideration of the boycott decision.

PEOPLE in the news

● DR. D. C. RHYS JONES, deputy director of the National Coal Board Research Establishment, has been appointed director in succession to Dr. J. Bronowski. Dr. Rhys Jones, who is 54, obtained his B.Sc. at Cardiff and Ph.D. at Manchester. He is an associate of the Institute of Chemical Engineering. After service with Scottish Dyes, I.C.I. and Powell Duffryn he joined the National Coal Board in 1944 as chief scientist in the South-Western Division and in September 1945 was transferred to the Coal Research Establishment as its deputy director.

● The programme of the International Congress on Pharmaceutical Legislation in European countries, held in Rome on 4, 5 and 6 May, included a report by MR. A. DUCKWORTH, secretary of the Association of British Pharmaceutical Industry.

● To mark 30 years' association with I.C.I. as a consultant PROF. A. M. J. F. MICHELS of Amsterdam University was presented with a gold watch by MR. J. C. SWALLOW, Plastics Division chairman. Prof. Michels became a consultant of I.C.I. in 1928 and was responsible for the design and construction of the first laboratory compressor for compressing gases to 3,000 atmospheres. It was with this machine that the world's first ton of polythene was produced by the Alkali Division in 1938.

● MR. R. B. SEYMOUR has been appointed associate director (manufacturing) of Aspro-Nicholas Ltd., Slough, Bucks. He has been promoted from production manager, a post which he held for 12 months following three years' service as technical manager. MR. L. COOMBS has been appointed associate director (home operations). His previous appointments with the company include that of supply manager and home sales manager.

● MR. FRANK CHAPMAN, M.A., has been appointed managing director of Carnegies of Welwyn Ltd., Welwyn Garden City, a company that was recently acquired by the Rexall Drug Co. Ltd., Loughborough, Leics.

● MR. CHARLES BERTRAM BROCKBANK, who has represented the Staveley Iron and Chemical Co. Ltd., Chesterfield, in North Wales for the past 31 years, retired in mid-May. Since 1939 he has worked from Colwyn Bay, but on his retirement the North Wales area will be served by the company's Manchester office, Grosvenor Chambers, 16 Deansgate.

● MR. H. H. BATES, chairman of Benzole Producers Ltd., was re-elected president for a second term at the sixth annual meeting of the National Benzole and Allied Products Association (N.B.A.). MR. F. A. JACKSON, managing director of Carless, Capel and Leonard Ltd., was re-elected vice-president. The executive committee was elected as follows: DR. M. BARASH (East Midlands Gas Board), W. S. DIXON (Dorman Long (Chemicals) Ltd.), C. M. FRITH (South Yorkshire Chemical Works), R. T. HAYES (United Coke and Chemicals), W. D. HOLT (East Midlands Division, N.C.B.), C. J. JOHNSON (North Thames Gas Board), J. E. JONES (South Eastern Gas Board), C. N. KEMP (South Western Tar Distilleries), M. J. R. LANE (Lincolnshire Chemical Co.), W. A. ROBINSON (Midland Tar Distillers), A. SLATER (Staveley Iron and Chemical Co.), C. J. WALLER (Scottish Tar Distillers). In accordance with a new rule MR. L. W. BLUNDELL (North Thames Gas Board) and MR. A. K. STEEL (National Coal Board) past presidents, were elected ex-officio members of the executive committee. The secretary is MR. W. E. CARTWRIGHT. Offices of the association are at 132 Sloane Street, London S.W.1.

● DR. W. BETTERIDGE has been appointed superintendent of the Platinum Metals Research Laboratory of the Development and Research Department, the Mond Nickel Company Ltd., Acton. Dr. Betteridge took a first-class honours degree in physics at Birmingham in 1931, obtained his Ph.D. in 1933 and his D.Sc. in 1958. After serving with Joseph Lucas Ltd., and the Bristol Aeroplane Company he joined the Mond Nickel Company in 1947. DR. G. L. J. BAILEY has been appointed superintendent of the Development and Research Department Laboratory in Birmingham. Dr. Bailey took a B.Sc.(Hons.) degree at Imperial College in 1937 and obtained his Ph.D. in 1939. After experience at the Admiralty Engineering Laboratory, West Drayton, he joined the British Non-Ferrous Metals Research Association, where he became deputy research manager. Since 1955 he has been superintendent of the Platinum Metals Research Laboratory at Acton. DR. W. STEVEN, superintendent of the Development and Research Department Laboratory in Birmingham, is being transferred to the Development and Research Division of the International Nickel Company, Inc., New York, as director of research. He has also been elected an assistant vice-president of that company.

Dr. Jonas Salk, the American discoverer of the anti-poliomyelitis vaccine, meets Dr. T. F. West, European operations executive of the African Pyrethrum Technical Information Centre, at the Royal Society of Health exhibition at Harrogate

● At the annual general meeting of the British Rubber and Resin Adhesive Manufacturers' Association on 5 May the following officers were re-elected: chairman, MR. D. E. CAMERON (B.B. Chemical Co. Ltd.); vice-chairman, MR. N. G. BASSETT SMITH (Dunlop Rubber).

● Sharples Centrifuges Ltd. have appointed MR. EDWARD C. C. HARVEY senior project engineer responsible for the company's activities in South-East England. Before joining Sharples Mr. Harvey was originally with the British Petroleum Co. Ltd. at their Abadan refinery and more recently with Shell Mex B.P.

E. C. C. Harvey

● SIR PAUL FILDES, formerly director of Chemical Bacteriology, Medical Research Council, on 9 May received an hon. degree of D.Sc. at Reading University.

● A Royal Society research studentship has been awarded to MR. K. L. MANCHESTER, M.A., of the Department of Biochemistry, University of Cambridge, to work on the mechanism of the regulation by hormones of protein synthesis at the Department of Biochemistry, University of Cambridge. A studentship has also been awarded to DR. M. L. WHELAN, M.A., Ph.D., of the Crystallographic Laboratory, Cavendish Laboratory, Cambridge, to carry out investigations of metals by transmission electron microscopy.

● Sheffield University has conferred the honorary degree of Doctor of Science on SIR HANS KREBS, F.R.S., Whitley Professor of Biochemistry at Oxford, formerly Professor of Biochemistry at Sheffield, and DR. RONALD HOLROYD, research director and a deputy chairman of Imperial Chemical Industries Ltd., who is a graduate of Sheffield.

● MR. R. B. ROBINSON has been elected chairman and MR. BRENDAN ROBINSON, vice-chairman, of the Midland Tar Distillers. MR. STANLEY ROBINSON, on medical advice, has relinquished the chairmanship but remains on the board.

● MR. H. O. JASPER and MR. F. GRUNWALD have been appointed to the board of Anglo-French Phosphate and MR. D. L. T. OPPÉ, MR. H. DEWEZ and MR. C. I. BALL have resigned their directorships. The secretary, MR. A. B. TOWNLEY, has resigned and MR. P. ROSE has been appointed to the position.

Commercial News

- **A.P.V. Chairman Reports Improved Position**
- **Pfeffer 60% Offer for United Indigo Accepted**
- **Staveley Working Profit Down £600,000**
- **Stafford Allen Double Their Capital**

Anglo-French Phosphate

Robert Benson Lonsdale and Co., together with certain other holders covering approximately 89% of the share capital of Anglo-French Phosphate have completed the sale of their shares to H. Jasper and Co. who will shortly make an offer to the remaining holders at the same price—18s 9d per 5s share.

The registered office has been changed to 103 Great Portland Street, London W.

A.P.V. Co. Ltd.

In the first three months of the current year, trading of the A.P.V. Co. showed an increase over the same period of last year. Mr. W. E. Jenkins, chairman, says this gives promise that the company's improved position will be maintained. In the past year extensive orders have been received for petrochemical plant for one of the largest refineries. In the export field, many new contracts were received for installation of plants in countries all over the world. Total export order business accounted for 44% of orders booked during 1958.

Beecham Group

In a plan for U.S. investors to take a bigger interest in shares of the Beecham group, the Morgan Guaranty Trust Company announce that they are prepared to accept deposits of these shares against which they will issue depository receipts in New York, where they will be traded on the "over-the-counter" market. The immediate result of this announcement was to raise Beecham 5s ordinary shares 1s 4½d to 33s 6d.

Cambridge Instrument

Application is being made for a quotation for the £1 ordinary shares of the Cambridge Instrument Company. Issued capital is £1,183,782 in ordinary and £27,670 in 7½ per cent cumulative preference £1 shares.

Profits after taxation and depreciation for the year ended 31 December 1958, were £221,547.

United Indigo and Chemical

The offer made by Pfeffer Brothers to acquire 60% of the preference and ordinary capital of the United Indigo and Chemical Company has been accepted by holders of the requisite number of shares.

A reduction in the capital of United Indigo and Chemical Co. Ltd., has been confirmed at the Lancashire Chancery Court. The company will now repay £47,500, a third of the paid-up capital—2s 6d on the 7s 6d preference and 8d on the 2s ordinary.

Staveley Iron and Chemical

A group working profit of £1,113,184 (£1,788,232) and net profit of £412,018 (£1,027,526) are announced by the Staveley Iron and Chemical Co. Ltd., a subsidiary of ISHRA, for the year ended 27 September 1958.

Dividends will take £230,000 (same).

German Potash Loan

The German Potash loan, raised in sterling by the selling syndicate of the German potash industry during 1925-29, is to be redeemed in full on or after 20 June, although the final maturity date is not until 1973. It is proposed to repay the bonds at £173 11s 5.15d per £100 nominal, plus 51 days' accrued interest.

Sandoz

Sandoz world turnover in 1958 reached a record of 503 million Swiss francs (about £41 million) compared with 488 million the previous year. This came from dyestuffs 184 million francs (189 million), pharmaceuticals 213 million (203 million), chemicals 106 million (96 million). Net profit was 16.8 million francs (16.6 million).

Dutch Chemical Companies

As is usual, financial reports have been issued by practically all the leading Dutch chemical companies in one week. Points from the reports are:

Algemene Kunstzijde Unie N.V.: Net profit for the first three months of 1959 was unchanged at 3.6 million guilders (£350,000). Turnover rose from 56.8 million guilders (£5.6 million) in 1957 to 61.4 million guilders (£6.05 million).

Chemische Fabriek Gembo N.V.: Net profit for 1958 was 276,000 guilders (£27,000) compared with 205,000 guilders (£20,000). Dividend is raised from 12% to 16%.

Koninklijke Nederlandsche Zoutindustrie N.V.: With the share of chemical products in the total output of 794,000 tonnes having risen 24% to 150,000 tonnes—the remainder being salt—dividend is 12% (same on lower capital). A new electrolysis plant has been brought in at Delfzijl.

New companies: Tanatex Chemical (Holland), in which Tanatex Chemical Corporation, Kearney, N.J., have an interest, will produce and distribute chemicals for the textile leather, paper and other industries. N.V. Olanda has been formed in Amsterdam to produce and distribute plastics and plastics raw materials and to obtain patents and licences to that end. Main interest is held by Allard and Co., N.V. Capital of both companies is 500,000 guilders (£48,000).

INCREASE OF CAPITAL

STAFFORD ALLEN AND SONS LTD., drug millers, oil pressers and distillers, etc., 20 Wharf Road, London N.1. Increased by £500,000, beyond the registered capital of £500,000.

APPOINTMENT OF RECEIVER

E. AND D. H. RICHARDS LTD., fertiliser manufacturers, Rednal Works, Rednal, Oswestry. R. F. Bendall, 126 Colmore Row, Birmingham, 3, has been appointed receiver and manager.

Market Reports

GOOD FLOW OF NEW ENQUIRIES

LONDON Steady trading conditions have been reported in the general chemicals market during the week. The leading industrial outlets are calling for good quantities against contracts, and there has been a sizeable flow of new home enquiry. Export trade is keeping up to recent levels. The demand for agricultural chemicals has eased now that the peak of the buying season has passed. Further fluctuations in zinc oxide prices have been notified, the prices operating from 11 May showing a reduction of £5 per ton; red seal is quoted at £90 10s per ton. Reasonably firm price conditions prevail in the coal tar products market.

MANCHESTER Prices on the Manchester market for heavy chemical products during the past week have maintained a generally steady front. The movement of supplies to consumers in the cotton and allied trades leaves plenty of room for improvement, but in most other directions delivery specifications against contracts are circulating reasonably well. Fresh enquiries from home users, as well as on export account, are on a fair scale. Quieter conditions are reported in the fertiliser market, but there is a quietly steady movement of the leading coal tar products, including creosote oil and the carbolic and cresylic acids.

GLASGOW The report from the Scottish heavy chemical market during the past week is one of reasonable activity, with requisitions against current and contract requirements well maintained. Demands were varied and quite a volume of enquiries were received.

Prices mostly remained firm with some slight variation taking place particularly relative to metal derivatives. A fair amount of activity can still be reported in regard to agricultural chemicals, both in demand and enquiries for seasonal requirements.

TRADE NOTES

Bonding Agents

Extensions of the range of Accobond bonding agents are described in a bulletin issued by Cyanamid of Great Britain Ltd., Bush House, Aldwych, W.C.2.

The originals were designed as anchoring agents for cellulose film when this was coated with nitrocellulose, polythene or Saran. Some uses of the new agents are: the anchorage of synthetic appliance enamel to steel, bonding cellulose nitrate lacquers to metals and glass, adhesion of acrylic latices to polyester film or cellulose acetate and bending and strengthening polyvinyl butyral/cotton or viscose rayon laminates.

Small-Scale Plant

Universities and colleges in the U.K. and overseas are among buyers for small-scale equipment made by Apex Construction Ltd., 15 Soho Square, London W.1.

The range has been extensively revised, and is described in leaflet SSE1.59. As far as is possible the items described are exact small-scale versions of large production sizes.

Controlling Algal Growth

Algal growth can be effectively and easily controlled by the use of Algistat, a product of the Laboratory Chemicals Division of the British Drug Houses Ltd., Poole, Dorset, it is stated in a new leaflet entitled 'Algistat'. The active constituent of the product is 2:3-dichloro-1:4-naphthoquinone. It has been extensively tested in both the U.K. and the U.S., and is said to have solved problems of algal growth in industry where trials have shown it unnecessary for regular and frequent plant 'shut-downs' for cleaning. One 18-oz. bottle of Algistat to every 50,000 gall. of water in the system is the recommended weekly dosage.

Hydrometers Prices Down

Details of the full range of hydrometers made by J. W. Towers and Co. Ltd., Victoria House, Widnes, are given in a new hydrometer catalogue, No. 709. The prices of many of the patterns shown have been reduced.

Non-toxic Bactericides

Technical bulletin E-5/1 from Armour Chemical Industries Ltd., 4 Chiswell Street, London E.C.1, gives details of Arquads. Arquad 16-50%, a quaternary-type bactericide (cetyl trimethyl ammonium chloride) has been used in hospital laundries to defeat outbreaks of infection of *Staphylococcus pyogenes* and among the many uses of Arquads are applications in sanitising rinses, air conditioning systems, food shops and bakeries.

Stafford Allen and Sons

Due to an error the name on an announcement in our Classified Advertisement section last week, p. 812, appeared as "Mr. A. W. Allen, Stafford and Sons Ltd." This should have read "Mr. A. W. Allen, Stafford Allen and Sons Ltd."

Lawn Fertiliser Applied in Solution

Boots the Chemists are marketing Lawn Reviver, a powder which is dissolved in water and is based on a formula containing nitrogen, soluble phosphoric acid, potash, and soluble iron (which helps to suppress moss).

N-Methyl Acetamide

British Celanese Ltd. have now extended the range of chemicals which they offer by including N-methyl acetamide at a minimum purity of 98.5%. This product has applications as a chemical intermediate, but it is of more particular interest in pharmaceuticals, for example as a solubilising agent for chloramphenicol. Inquiries should be addressed to chemical sales department at 345 Foleshill Road, Coventry.

Leek Chemicals and Hoechst

Leek Chemicals Ltd. announce that negotiations between Farbwerke Hoechst and themselves for the marketing in the United Kingdom of Hoechst plant protection products have been discontinued by mutual consent.

New Telephone Number

From 9 June the telephone number of the Reed Paper Group companies at the Aylesford site, near Maidstone, Kent, will be Maidstone 7-7777. Companies to be reached through the new number include Reed Corrugated Cases Ltd. (New Hythe branch) and Medway Paper Sacks Ltd.

Grinding Machinery

Ball mills, tube mills and rod mills are described in a leaflet on grinding machinery by Stockton Forge Ltd., Stockton-on-Tees, Co. Durham, a Head Wrightson company.

Ball mills, in closed circuit with Akins classifiers, are suitable for the wet grinding of ores in flotation and for cyanidation plants. Single stage grinding requires the grate-discharge type of ball mill while grate and overflow discharge type mills are best suited to primary and secondary duties respectively in double stage grinding.

Tube mills may be used instead of ball mills as secondary grinders in cyanidation plants. Rod mills are preferable for the smaller reduction range and for the granular product required in jig and table concentration plants.

Basically all three kinds of mill consist of a welded mild steel tube provided with renewable wearing liners and a grinding medium. The tube rotates on its axis while material enters one hollow trunnion and is discharged in a reduced state at the other end.

Dial Thermometers

A range of new mercury-in-steel (Mersteel) rigid stem-dial thermometers is described in publication T/47 by Negretti and Zambra Ltd., 122 Regent Street, London W.1.

DIARY DATES

WEDNESDAY 20 MAY

Royal Inst.—London: 21 Albemarle Street, W.1, 9 p.m. 'The molecular basis of life', by Dr. M. F. Perutz.

Royal Statistical Soc.—London: School of Hygiene and Tropical Medicine, W.C.1, 5.15 p.m. A discussion on simulation studies of industrial operations. Mr. P. V. Youle, Dr. K. D. Tocher, Mr. W. N. Jessop and Mr. F. I. Musk.

S.A.C.—London: Burlington House, Piccadilly, W.1, 7 p.m. 'The determination of Vitamin E: a collaborative study of quantitative paper chromatography and the development of a recommended method of analysis', by Mr. A. L. Bacharach, Dr. J. Green, Dr. P. W. Russell Eggitt and Mr. R. J. Ward.

FRIDAY 22 MAY

C.S.—Exeter: Washington Singer Laboratories, 5 p.m. 'The acetylenic approach to the synthesis of natural products', by Prof. R. A. Raphael.

Soc. of Cosmetic Chemists—London: 55 Park Lane, W.1, 7 p.m. A.g.m.

Properties of Acetylene in Welding

MONSIEUR P. PHILIPPON, former director of the Société de l'Air Liquide, proposing the toast of the British Acetylene Association at the annual luncheon of the association on 6 May, said it was the oldest among the acetylene associations still in existence.

It was important, M. Philippon said, to spread as far as possible the safety rules for their industry and avoid accidents which might have a grievous influence on the whole acetylene industry. Acetylene, he thought, had already been brought into disrepute by promoters of liquefied gases like propane and butane, who recommended their products specially for oxy-cutting. It was the duty of their associations to draw attention to the fact that the oxy-acetylene flame had properties which allowed performances not possible with combustibles other than acetylene.

Mr. E. Seymour-Semper, managing director, Hancock and Co. (Engineers) Ltd. and president of the association, responded to the toast.

£8,000 for Man Injured at I.C.I. Nobel Works

GEORGE F. LENNON (38), process worker, of Stevenston, Ayrshire, who lost a leg and sustained other serious injuries as a result of an explosion at the Ardeer factory of Imperial Chemical Industries, Ltd., in March 1954, was awarded £8,800 damages by a jury sitting with Lord Guthrie in the Scottish Court of Session.

Lennon, who sued the company for £14,000, complained that as a result of very extensive burning and other injuries he had suffered a serious psychological upset. He said he was fit for only limited work.

The accident resulted from the bursting of a disused pipe caused, it was said, by the generation of heat through decomposition of acid refuse.

John Blake (32), of Ardrossan, who was injured in the same explosion, and sued for £6,000, was awarded £3,500.

I.C.I. admitted liability in both actions, but contested them on the ground that the sums claimed were excessive.

NEW PATENTS

By permission of the Controller, HM Stationery Office, the following extracts are reproduced from the 'Official Journal (Patents)', which is available from the Patent Office (Sales Branch), 25 Southampton Buildings, Chancery Lane, London W.C.2, price 3s 6d including postage; annual subscription £8 2s.

Specifications filed in connection with the acceptances in the following list will be open to public inspection on the dates shown. Opposition to the grant of a patent on any of the applications listed may be lodged by filing patents form 12 at any time within the prescribed period.

ACCEPTANCES

Open to public inspection 24 June

Silicon tetrachloride production. Monsanto Chemicals Ltd. **815 276**

Photosensitive addition polymerisable compositions and products made therefrom. Du Pont de Nemours & Co., E. I. **815 277**

Nitriles and amides of pyridine carboxylic acids. Aries Associates Inc. **815 278**

19-nor-delta-4-Androsten-3-one-17-β-ol hexahydrobenzoate and method for manufacturing same. Hicks, L. **815 341**

Colouring of hair. Ashe Laboratories Ltd. **815 346**

Quaternary pyridylisoquinoline derivatives. Imperial Chemical Ltd. **815 348**

Preparation of filled papers and the like. Rohm & Haas Co. **815 410**

Preparation of peracetic acid. Shawinigan Chemicals Ltd. **815 353**

Method of producing dehydrated orange juice. Vacu-Dry Co. **815 354**

Heat-resistant lubricant. Phoenix-Rheinrohr Aktiengesellschaft Vereinigte Hütten-und Röhrenwerke. **815 355**

Process and apparatus for producing sheets of thermoplastic material. Pedersen, B. H. **815 413**

Silicon carbide resistance bodies and methods of making same. Carborundum Co. **815 357**

Vitamin E fortified animal feeds. Eastman Kodak Co. **815 414**

Manufacture of derivatives of polysaccharides, more particularly of starch, by a dry process. Scholten's Chemische Fabrieken N.V., W. A. **815 358**

Production of N-nitroso-N-alkyl- and N-nitroso-N-cycloalkyl-hydroxylamines. Badische Anilin- & Soda-Fabrik A.G. **815 537**

N-nitroso-hydroxylamines and fungicidal agents. Badische Anilin- & Soda-Fabrik A.G. **815 538**

Method of producing zirconium oxide from zirconium tetrachloride. Goldschmidt A.G., T. **815 425**

Production of tetracyclines. American Cyanamid Co. [Divided out of 815 524.] **815 525**

Catalytic process for polymerisation of olefinically unsaturated hydrocarbons and catalysts for use therein. Petrochemicals Ltd. **815 429**

Stable solution for producing ice colours. General Aniline & Film Corp. **815 458**

Oxo process for the preparation of oxygenated organic compounds. Union Carbide Corp. **815 566**

Benzomorpholone derivatives and a process for the manufacture thereof. Farbenfabriken Bayer A.G. **815 279**

Preparing 3-methyl-3-carboxycyclohexanone-(1)-2 β-propionic acid and derivatives thereof. Abildgaard, K. [trading as Lovens Kemiske Fabrik Ved. A. Kongsted]. [Addition to 803 426.] **815 567**

Method of producing nitro-starch. Wasag-Chemie A.G. **815 280**

Method of producing photostable phenols. Metallgesellschaft A.G. **815 568**

Silicon steel with improved magnetic anisotropy and method of making same. General Electric Co. **815 569**

Compositions for controlling plant growth. Bataafsche Petroleum Maatschappij N.V., De. **815 570**

Gas furnace for carrying out gas reactions in ceramic tubes. Deutsche Gold Und Silber Scheideanstalt Vorm Roessler. [Divided out of 815 530.] **815 531**

Polymerising olefinic hydrocarbons. Phillips Petroleum Co. [Divided out of 815 520.] **815 521**

Open to public inspection 1 July

Production of plutonium 239. Brown, H. S. and Anderson, H. L. **815 641**

Manufacture of titanium dioxide. Laporte Titanium Ltd. **815 891**

Resilient materials. Hairlok Co. Ltd. **815 898**

Heat-treatment furnaces. Dowson & Mason Ltd., formerly Dowson & Mason Gas Plant Co. Ltd., and Leonard, L. G. A. **815 899**

Polyesters. Farbwerke Hoechst A.G. **815 902**

Polyazo-dyestuffs derived from 3, 3^1-dihydroxydiphenylamine and processes for their manufacture. Farbwerke Hoechst A.G. **815 737**

Dyeing textile materials of linear aromatic polyesters. Farbwerke Hoechst. **815 738**

Lake-like brightening agents Farbenfabriken Bayer A.G. **815 742**

Cosmetic preparations for the hair. Ciba Ltd. **815 784**

Quaternary ammonium compounds and preparation thereof. Rohm & Haas Co. **815 745**

Manufacture of dioxy-nitro-arylaminoanthraquinones and the products obtained thereby. Ciba Ltd. **815 746**

Apparatus for producing intimate contact between a liquid and a gas. Ajem Laboratories, Inc. **815 592**

Liquid-vapour barrier compositions and articles made thereof. Goodrich Co., B F. **815 907**

Densimetric separation of solid materials. Douy, A. **815 594**

Production of electrolytic metal coatings. Dehydag Deutsche Hydrierwerke G.m.b.H. (Addition to 804 403). **815 908**

Porous metal wall construction and method of manufacture. California Institute Research Foundation. **815 596**

Extracting phenols from phenolic oils. Veb Leuna-Werke W. Ulbricht. **815 754**

Coating surfaces and, particularly, metal surfaces. Wamant, G. and Tartinville, J. **815 756**

Rapid cooling of the reaction product gases in catalytic oxidation processes. Veb Leuna-Werke W. Ulbricht. **815 600**

Production of agglomerated bodies using aluminium nitride. Pechiney Compagnie de Produits Chimiques et Electrometalluriques. **815 602**

Subliming process carried out in a bed of fluidised solid. Farbenfabriken Bayer A.G. **815 603**

Sublimation of substances in a fluidised bed sublimer. Farbenfabriken Bayer A.G. **815 604**

Method and apparatus for generating heated gases. Combustion Engineering Inc. **815 724**

Epicyclic tranmission. Fichtel & Sachs A.G. **815 913**

Conversion of cellulose derivatives into granular form. Olin Mathieson Chemical Corp. **815 788**

Metallisable benzene-monoazo-benzene dyestuffs, complex heavy metal compounds thereof, and their use. Geigy A.G., J. R. **815 790**

Production of crystals from solutions. Power Gas Corp. Ltd. **815 791**

Manufacture of cellulose esters. British Celanese Ltd. **815 792**

Production of electrolytic metal coatings using internal salts of aromatic bases as plating bath additives. Dehydag Deutsche Hydrierwerke G.m.b.H. **815 916**

Coating compositions containing metal particles. American Marietta Co. **815 793**

Recovery and purification of vitamin B_{12} concentrates. Aschaffenburger Zellstoffwerke A.G. (Addition to 748 043) **815 917**

Cellulose ester textile fabrics. Celanese Corp. of America. **815 794**

Electrolysis cell. National Distillers & Chemical Corp. **815 922**

Rotary Apparatus. U.K. Atomic Energy Authority. **815 923**

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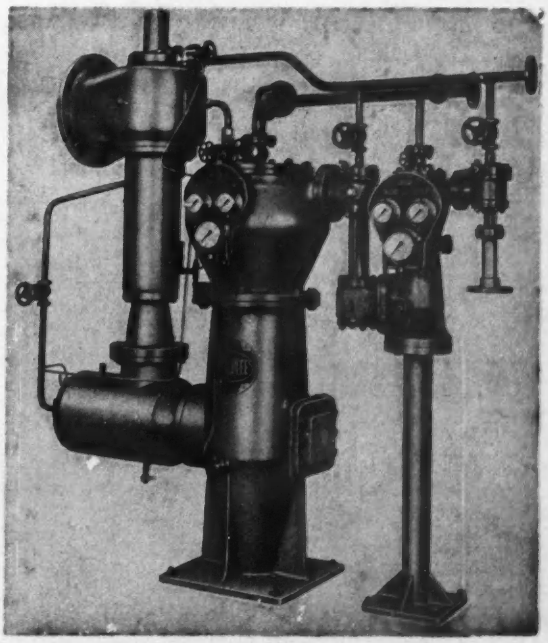

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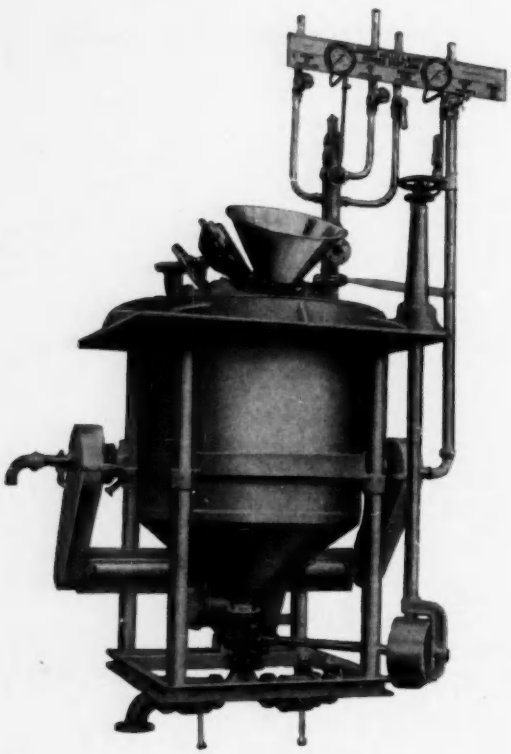

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